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The Journal *of the* SOCIETY OF DYERS AND COLOURISTS

Volume 68 Number 3

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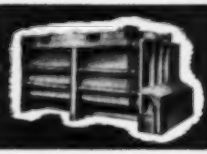

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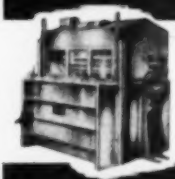

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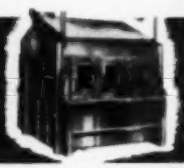

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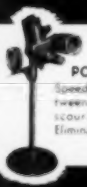

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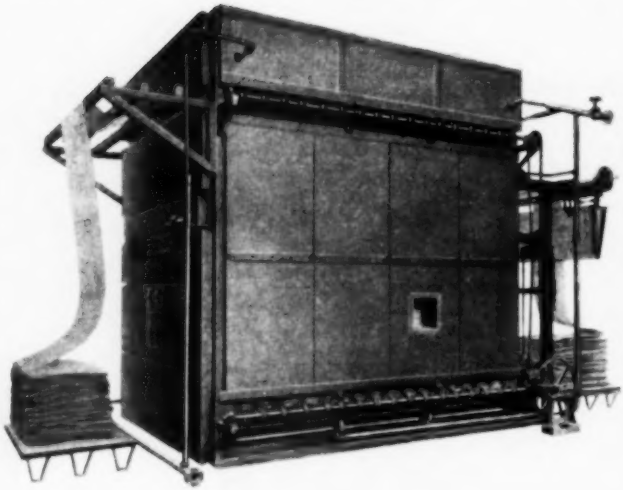

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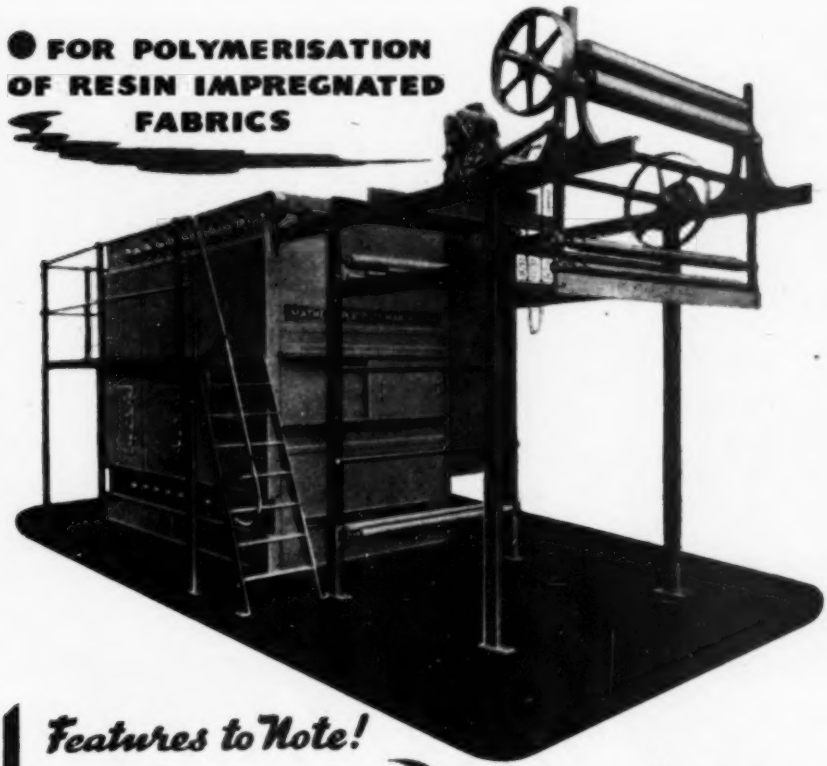

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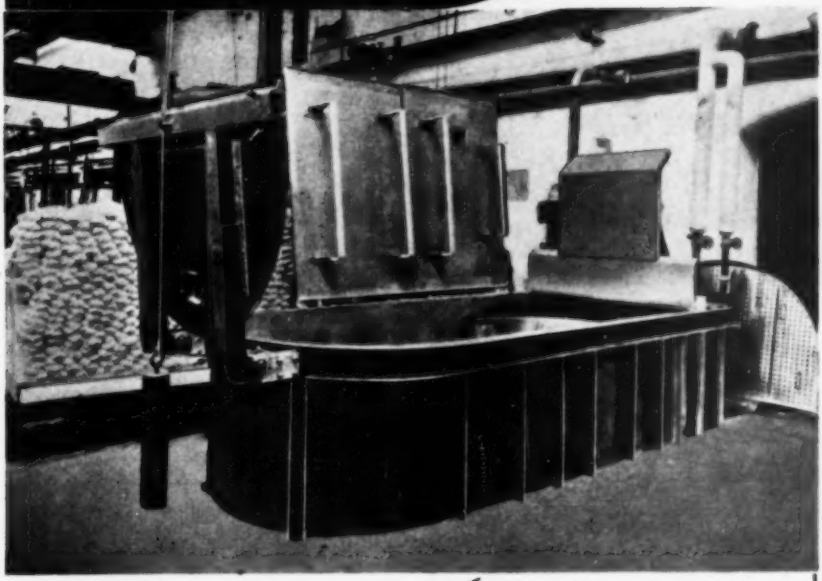

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NOTICE TO MEMBERS AND SUBSCRIBERS

Readers requiring *general information* regarding the Official Notices, List of Officers of the Society, etc. should consult pages 1–6 of the January 1952 and pages 253–256 of the July 1951 issues of the *Journal*, or write to *The General Secretary*, The Society of Dyers and Colourists, 19 Piccadilly, Bradford, Yorkshire (*Telephone* Bradford 25138–9). *Editorial Communications* should be addressed to *The Editor*, at the same address.

Forthcoming Papers

The following papers have been accepted by the Publications Committee, and will appear in future issues of the *Journal*—

COMMUNICATIONS

Package Dyeing—
I—A Theoretical Model and its relation to Technical Practice — *J. Boulton and J. Crank*

The Properties of Some Derivatives of Indanthrone and Flavanthrone — *W. Bradley, H. E. Nürsten, and E. Leek*

A New Method for the Dyeing of Mineral Khaki — *E. H. Daruwalla and G. M. Nabar*

MEMBERS' CHANGES OF ADDRESS

Alexander, Dr. P., formerly of South Kensington, to The Chester Beatty Research Institute, The Royal Cancer Hospital, Fulham Road, London S.W.3.

Arneson, Finn, formerly of Norway, to 41 Thurlington Road, Narborough Road, Leicester.

Beveley, A. F., formerly of Port Elizabeth, to P.O. Box 1519, Cape Town, S. Africa.

Borten, S., formerly of Caulfield, to 378–380 High Street, St. Kilda, S2, Victoria, Australia.

Cox, N., formerly of Accrington, to Hycroft, Shelling Hill, Cullybackey, Co. Antrim, Northern Ireland.

Daitz, V., formerly of Durban, to c/o Natal Cotton & Woollen Mills Ltd., P.O. Box 17, Merebank, Natal, S. Africa.

Dalton, A. B., formerly of P.O. Box 2929, to 203 Senior Drive, Northcliff, Johannesburg, South Africa.

Eckersall, W., formerly of Birtle, to 119 Manchester Road, Walmersley, Bury, Lancs.

Faulkner, J., formerly of 4 Fir Grove, to 53 Garden Village, Muckamore, Co. Antrim.

Pitton, J., formerly of Huddersfield, to The Woodlands, Woodbottom Lane, Shead Syke, Brighouse.

Fletcher, R. E., formerly of Blackley, Manchester, to c/o Dyehouse Dept., Clayton Aniline Co. Ltd., Clayton, Manchester.

Grindley, F. D., formerly of N. Ireland, to 143 Withins Lane, Breightmet, Bolton.

Hargreave, A. G., formerly of London E.11, to 35 Holmwood Road, Cheam, Surrey.

Inman, E. R., formerly of Cumberland Road, to 15 Headingley Avenue, Leeds 6.

Johnstone, W. H., formerly of Addison Road, to 135 Castleton Road, Deepdale, Preston.

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Redston, R. W., formerly of Montreal 36, to 75 Highfield Avenue, Town of Mount Royal, Montreal 16, Quebec, Canada.

Reed, F. G., formerly of 29 St. Patrice, to 353 St. Patrick West, Magog, P.Q., Canada.

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Smith, S., formerly of 125 Broadway, to 721 Moston Lane, Moston, Manchester 10.

Speke, R. W., formerly of Manchester 20, to West Lea, 163 Manchester Old Road, Middleton, Lancs.

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Taylor, John, formerly of Hong Kong, to c/o I.C.I. Ltd., Dyestuffs Division, Overseas Sales Dept., Hexagon House, Blackley, Manchester 9.

Weissink, J. L., formerly of Eindhoven, to J. H. W. Roberstr. 68, Enschede, Holland.

Wild, J., formerly of N. Ireland, to c/o 63 Leyland Lane, Leyland, Lancs.

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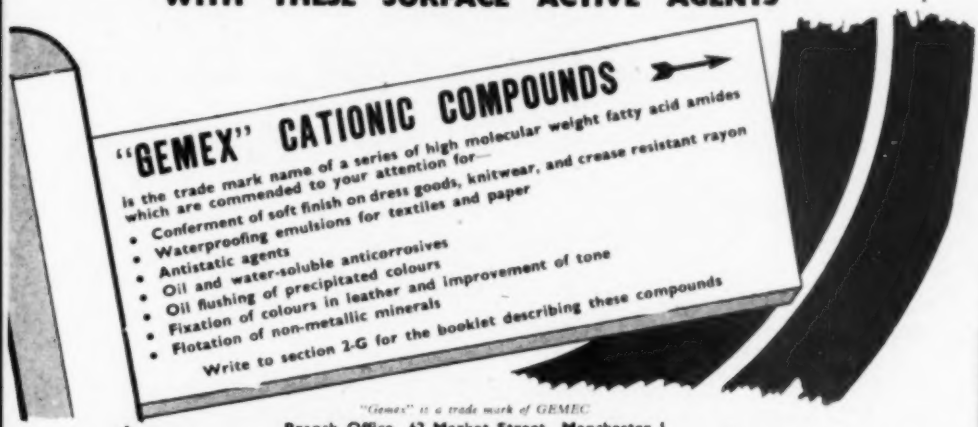
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Forthcoming Meetings of the Society

Friday—28th March 1952
ANNUAL GENERAL MEETING and DINNER of the Society Queens Hotel, Leeds

Friday—9th May 1952
Ninth Mercer Lecture Dr. F. C. Wood *Non-felting Wool and Wool Mixtures* College of Technology, Leicester

HUDDERSFIELD SECTION

All meetings held at Field's Café, Huddersfield at 7.30 p.m., unless otherwise stated.

1952

Tuesday 8th April — ANNUAL GENERAL MEETING
Dr. G. H. Lister (Sandoz Products Ltd.) *Acid and Chrome Dyes on Wool*

MANCHESTER SECTION

1952

Friday 18th April — ANNUAL GENERAL MEETING
R. Thornton, Esq., M.Sc. *Impressions of the Printing Industry Overseas.* Gas Showrooms, Town Hall Extension, Manchester. 6.30 p.m.

MIDLANDS SECTION

All meetings commence at 7 p.m.

1952

Wednesday 26th March — G. H. Lister, Esq., Ph.D., B.Sc. *The Influence of Dyeing Methods on Fibre Damage.* College of Technology, Leicester

Wednesday 23rd April — G. K. Mecklenburgh, Esq., S. Shaw, Esq., and H. W. Peters, Esq. *Some Developments in Setting and Finishing Nylon Textiles* (Preceded at 6.45 p.m. by the Annual General Meeting of the Section). King's Head Hotel, Loughborough

NORTHERN IRELAND SECTION

1952

Saturday 5th April — ANNUAL GENERAL MEETING and DINNER
Queen's Hotel, Belfast
A.G.M. 5 p.m. Dinner 6.30 for 7 p.m.

BRADFORD JUNIOR BRANCH

All meetings held in the Bradford Technical College at 7.15 p.m.

1952

Tuesday 25th March — E. E. Tallis, Esq., B.Sc., A.R.I.C. (Courtaulds Ltd.) *Recent Developments in Viscose Rayon and Staple Manufacture*

There will be a visit to Salts (Saltaire) Ltd., the date of which has not yet been fixed.

WEST RIDING SECTION

All meetings held at the Great Northern Victoria Hotel, Bradford, at 7.15 p.m. unless otherwise stated

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Thursday 27th March — ANNUAL GENERAL MEETING

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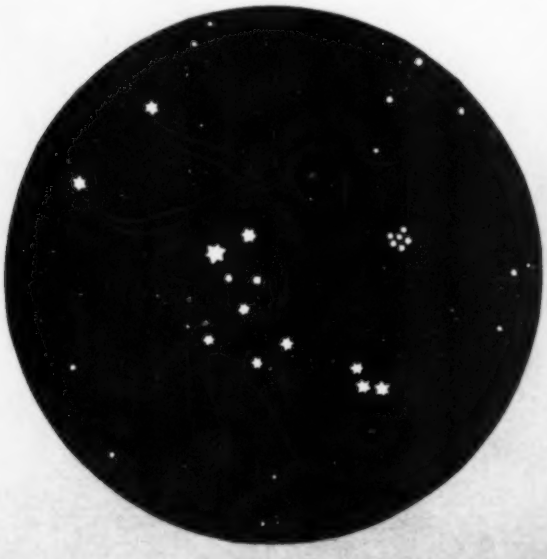

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THE JOURNAL
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Volume 68—Number 3 MARCH 1952 Issued Monthly

His late Majesty King George VI

With sorrow we record the death of His Majesty King George VI, in whom we of his peoples have lost our king and the head of our family of nations, and the world has lost a good man who did his duty.

To Queen Elizabeth the Queen Mother, the Queen, and Princess Margaret we send our sympathy on their loss of a devoted and beloved husband and father, which sympathy also goes to Queen Mary on the loss of a son whose career can have given her nothing but joy and satisfaction. For ourselves may our memory of him always inspire us to emulate that dogged devotion to duty he displayed throughout life.

To his daughter and successor, Her Majesty Queen Elizabeth II, Supreme Lady of the British Empire and Head of the Commonwealth, who has already dedicated her life to our service, we pledge the same unswerving loyalty that we gave to her father, and our unsparing efforts to make her reign peaceful, prosperous and glorious.

COMMUNICATIONS

Studies in the Fundamental Processes of Textile Printing
IV—The Transfer of Acid Dyes to Cellulose during Steaming

R. B. Patel and H. A. Turner

A selection of acid dyes, including representatives of the group suitable for direct printing on regenerated cellulose, has been examined by the method of Munshi and Turner [1]. It is found that the transfer of some of them from starch to cellulose during steaming passes through a maximum as steaming continues, and also that the exhaustion of the starch layer under standard conditions increases with increasing initial concentration of dye in the starch. The effect of a number of assistants, including sodium chloride and urea, has been examined. It seems probable that the distribution of the dye between starch and cellulose is closely connected with the distribution of water between these two phases.

Introduction

ACID DYES IN DIRECT PRINTING

About 1935 it was observed that Brilliant Cyanine 6B (Ciba) could be applied to fabrics of regenerated cellulose fibres by a direct printing method without mordanting. The uptake of the dye was assisted by adding thiodiglycol or resorcinol to the printing paste. The prints so obtained had the brightness characteristic of many acid dyes, and the fastness to water and washing, while not very high, was sufficient for many purposes. Since then, a number of other acid dyes have been added to the printing range, and urea is a common constituent of the printing pastes which are used. Other additions that have been recommended include organic liquids, such as thiodiglycol and pyridine, which are recognised as powerful solvents for many classes of dyes, soluble inorganic phosphates, and sodium chlorate, the last, presumably, as a protection for the dye against reduction during steaming. Not all acid dyes are suitable. Soluble levelling dyes of low molecular weight, such as Acid Magenta or Tartrazine, are not fixed in useful proportions, nor are the fast milling dyes.

The exhaustion of the dye from the applied print to the cellulose during steaming depends on the conditions within the steamer. Meitner observed for Brilliant Cyanine 6B that transfer to cellulose in the absence of any assistant was satisfactory if the steam were wet. If the steam were dry, then it was necessary to add urea. Saville [2] showed that the presence of such assistants as ethylene glycol shortens the steaming time necessary. du Pont [3] have concluded that urea is indispensable as a general assistant in printing this style.

Meitner [4] first subjected the printing mechanism

of acid dyes to a quantitative examination. He measured the standard rate of diffusion, in aqueous solution, of a number of acid dyes and thence calculated the mean particle diameter at room temperature, this quantity being assumed to represent the relative tendencies of the different dye molecules to aggregate. The dyes with extremes of high and low particle size were alike shown to be unsuitable for printing, the best printing dyes being therefore those with a medium tendency to molecular aggregation. A theory was evolved in which it was assumed that the printing dyes formed polydisperse systems in water and, during steaming, could break down progressively into particles sufficiently small to diffuse out of the thickener layer and into the water-filled pores of the cellulose. Once within, reaggregation of the molecules was assumed, leading to the retention of the dye. The more highly dispersed members of the series, lacking this power of reaggregation, diffuse out of the fibre almost as quickly as they diffuse in; the more highly aggregated are unable to disperse into a sufficient number of particles small enough to enter the fibre in the first place, and so cannot dye it very deeply. It will be seen that a mechanism of this kind is likely to be affected by the amount of water in the system during the transfer of the dye from one phase to the other, and therefore by the presence of substances, such as urea, which can be regarded either as increasing the dispersion of the dye itself or as causing enhanced swelling of the thickener or the fibre during steaming.

COMPARISON OF ACID AND DIRECT DYES

As already suggested in previous papers [1,5], application of the direct dyes by printing, because it presents the dye to the fibre at a relatively high concentration, and perhaps also because it modifies favourably the electrical conditions at the fibre surface, is able to mitigate some of the factors which oppose diffusion of the dye into the cellulose and its fixation therein by valency forces acting between dye and fibre molecules. Only a few acid dyes (e.g. Coomassie Red PGP) show any marked affinity for cellulose of the kind shown by the direct cotton dyes, and therefore are absorbed in any useful amount from solutions of the order of concentration normally used for dyeing from aqueous baths. As seen above, however, they can be applied by a printing method. It was natural therefore to attempt to extend the investigation commenced with the direct cotton dyes to the acid dyes. This has now been done, and, as a result, it is shown that typical acid printing dyes have transfer properties on steaming which distinguish them sharply from the direct cotton dyes. Some of these properties are consistent with the theory put forward by Meitner [4].

SUMMARY OF RESULTS

(1) Four dyes were examined for the course of transfer, during steaming, from a film of starch (farina) to a film of regenerated cellulose (Cellophane) in contact with it over a standard area. These were—(*a*) Naphthalene Scarlet R, low in the Meitner series of particle size, and given by him the value 7·1 A. for mean particle radius; (*b*) Coomassie Brilliant Blue FF (particle radius 22·5 A.), a dye of good technical printing properties; (*c*) Coomassie Brilliant Blue R (63·2 A.), which is, according to Meitner, near the upper limit of particle size in respect of suitability for printing on viscose, but which is very suitable for printing on cuprammonium rayon; and (*d*) Carbolan Yellow 3G (66·8 A.), which has a low exhaustion on printing. In all cases the dyes were freed from electrolytes and other impurities by repeated crystallisation from water.

(2) With transfer under standard conditions, in the absence of assistants, the exhaustions were essentially in the order predicted by Meitner, viz. Coomassie Brilliant Blue FF, Coomassie Brilliant Blue R, Naphthalene Scarlet R, and Carbolan Yellow 3G in descending order.

(3) With the first and third members of the above series, migration of dye from starch to cellulose takes place in the first part of the steaming, reaching a maximum for Naphthalene Scarlet R in 10 min. and for Coomassie Brilliant Blue FF in 60 min. As steaming continues, part of the dye first transferred to the cellulose returns to the starch (Fig. 1 and 3). Coomassie Brilliant Blue R does not appear to undergo this reverse migration, and in time approaches a condition of equilibrium exhaustion (Fig. 9).

(4) The exhaustion from the starch of Coomassie Brilliant Blue FF and, to a less pronounced extent, of Naphthalene Scarlet R increases with increasing initial concentration of dye in the starch film. This is unusual behaviour, since with most substantive dyes, exhaustion diminishes with increasing concentration in the dyebath.

(5) In general, the presence of urea or of common salt tends to cause a reversal of migration earlier in the steaming, and to diminish the maximum value for the exhaustion. The effect varies markedly with change in the concentration of urea. Addition of urea causes the transfer of Coomassie Brilliant Blue R to pass through a maximum also.

(6) Transfer of Coomassie Brilliant Blue FF can be accomplished without the application of heat if a starch paste containing the dye is spread on the surface of cellulose sheet and is dried at room temperature over a period of several hours. The slower the rate of drying, the slower the transfer.

On the basis of these observations, it is suggested that the distribution of dye between starch and cellulose is connected with the relative water contents of the respective phases during the course of the steaming.

Experimental

MATERIALS

(*I*) *Dyes*

NAPHTHALENE SCARLET R (ICI)—This was prepared from a commercial sample by repeated crystallisation, by cooling, from hot concentrated aqueous solutions and, in the later stages of the purification, from ethanol–water mixtures, until after drying at 110°C. for 12 hr. samples from two

successive crystallisations gave identical readings with the Spekker absorptiometer when examined at standard concentration in 20% pyridine–water solution.

COOMASSIE BRILLIANT BLUE FF (ICI) and COOMASSIE BRILLIANT BLUE R (ICI)— Commercial samples of each of these dyes were first crystallised by cooling from hot saturated aqueous solutions. With the removal of the impurities they tended to separate as gels. They were therefore isolated, dried, and then extracted with water in a Soxhlet until about 25% of the weight of the dry product had been removed. The solid material remaining was then dried, and extracted with dry ethanol until a residue of about 25% remained. The residue was discarded, and the alcoholic extract was evaporated to dryness. The above series of operations was then repeated until samples from two successive purifications gave identical Spekker readings when examined at standard concentration.

CARBOLAN YELLOW 3G (ICI)— This was crystallised five times from a boiling saturated solution. In the last three stages it was necessary to cool the solution by surrounding with ice. Samples from the last two crystallisations gave identical Spekker readings when examined at standard concentration.

(2) *Thickeners*

STARCH— A good-quality farina, of ash content (by ignition with concentrated sulphuric acid) 0·27%. This thickener and the reasons for its use have been discussed in a previous paper [5].

GUM TRAGACANTH— A good-quality, powdered white tragacanth (T. S. Whittle Ltd.).

SODIUM ALGINATE—Manucol V (Albright & Wilson), a high-viscosity, foodstuff grade.

METHYL CELLULOSE— Colloresin DK (IG), commercial.

(3) *Assistants*

SODIUM CHLORIDE and UREA— AnalaR.

GLYCEROL— *B.P.*

PYRIDINE— Redistilled, b.p. 114–115°c.

CETYLPYRIDINIUM BROMIDE— Fixanol (ICI), commercial.

(4) *Cellulose Sheet*

CELLOPHANE (600 PT grade) was soaked in repeated changes of distilled water for 24 hr., and preserved in distilled water until required for use. Thickness of soaked material 0·082 mm.; weight per sq. cm. 4·704 mg. (soaked).

PREPARATION OF DYE-CONTAINING STARCH FILM

The procedure has already been described in detail [1]. The thickness of the film when dried and conditioned at 75% R.H. was 0·09–0·1 mm. It was not always possible, because of the intense colour of the film, to estimate the dye content by measurement of the optical density of the dry material. The dye contents of both starch and cellulose after steaming and separation were determined on extracts of the respective films with 20% pyridine, and the exhaustions calculated from these values.

STEAMING, ETC.

The procedure for cutting discs of starch film ($\frac{11}{16}$ in. in diameter), adhesion to soaked cellulose sheet, steaming, separation, extraction of dye from each phase, and colorimetric determination have followed the lines previously described. Because of the very high mobility of some of the dyes within the system, it was necessary to commence steaming immediately after starch and cellulose had been brought into contact, and to separate the films without delay on removal from the steamer.

Difficulty was experienced in handling some of the starch films containing the higher concentrations of urea, which separated on drying as a network of interlacing crystals upon and within the dry film, making it uneven and brittle.

EXPERIMENTS WITH DRIED-ON FILMS OF STARCH

For these experiments, owing largely to the interference of the urea noted above, a sheet of water-soaked cellulose film was squeegeed into uniform contact with a glass plate, and the dye-containing paste was spread upon the cellulose. This was then dried down at room temperature in a circulated atmosphere of the desired relative humidity. The thickness of the final dry layer of starch was, of course, controlled by the original thickness of the layer of paste and its thickener content. Strips of this composite film were steamed, and, after removal from the steamer, were cut into discs of the standard size before the two layers were separated and the dye in each was estimated.

Experimental Results

The results will first be arranged under separate headings for each dye examined.

NAPHTHALENE SCARLET R

The transfer of the purified dye at three different initial concentrations in the starch without the addition of assistants is shown in Fig. 1. It will be

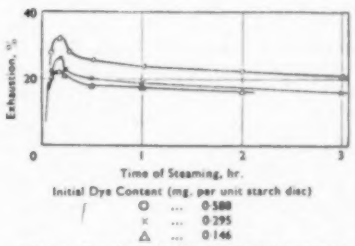


FIG. 1— Transfer of Naphthalene Scarlet R pure to Cellulose during Steaming

seen that— (*a*) the highest exhaustion attained is 33%; (*b*) a large proportion of the total transfer to cellulose has taken place in the first 5 min. of steaming; (*c*) this transfer reaches a maximum value in approx. 10 min. from the commencement of the experiment; and (*d*) in general, the higher the initial concentration of dye in the starch, the greater the transfer to the cellulose.

Transfer at one initial concentration of dye is shown (Fig. 2) when additions of sodium chloride and of urea have been made to the paste. Both these assistants cause a general lowering of the

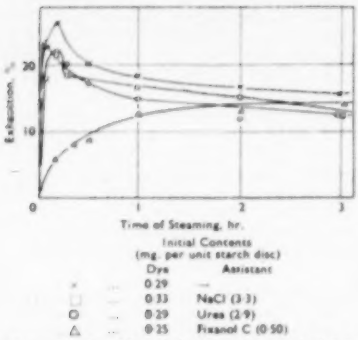


FIG. 2.—Transfer of Naphthalene Scarlet R pure to Cellulose during Steaming in the presence of Assistants

exhaustion, and the urea causes the maximum exhaustion to be reached more rapidly.

It was thought that the small particle size of this dye, which should allow it to diffuse very rapidly in the starch and cellulose layers, and which should, according to Meitner, be responsible for its relatively low exhaustion, might be favourably modified by the addition of a cationic fixative. This, by association with the dye molecules, should increase the size of the migrating units, and, perhaps, lead to greater retention of dye in cellulose. Fixanol C was the agent used. It will be seen (Fig. 2) that this retards the dye transfer during the early part of the steaming, and suppresses all signs of a maximum, although ultimately the exhaustion equals that when no assistant is used. The weight of Fixanol which could be used was limited to about twice that of the dye present. Larger amounts caused softening and irregularity in the texture and thickness of the film.

COOMASSIE BRILLIANT BLUE FF

The transfer of this dye at a number of initial concentrations in the starch is shown in Fig. 3. From this it is seen that—(*a*) the initial rate of transfer is lower than with Naphthalene Scarlet R; (*b*) exhaustion increases progressively with increase in initial concentration in the starch; (*c*) after an initial concentration of about 0·06 mg. of dye per standard disc has been exceeded, a maximum value for exhaustion is reached in about 1 hr. steaming; and (*d*) the general exhaustion for the same initial concentration is higher than with Naphthalene Scarlet R.

Taking those curves in which a maximum occurs, and plotting from them the weight of dye transferred to the cellulose after 1 hr. against the weight of dye originally in the starch, the straight line in Fig. 4 results. This curve, if produced, will cut the dye-in-starch axis at a value of 0·085 mg.

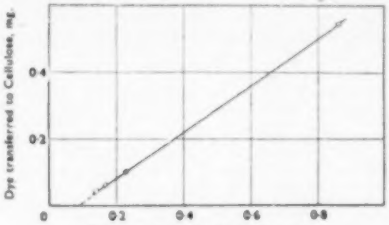


FIG. 4.—Transfer of Coomassie Brilliant Blue FF pure after 1 hr. Steaming

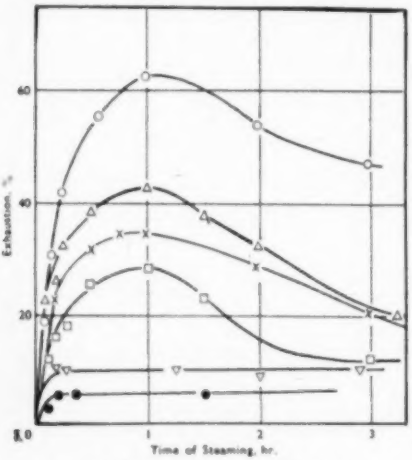


FIG. 3.—Transfer of Coomassie Brilliant Blue FF pure to Cellulose during Steaming

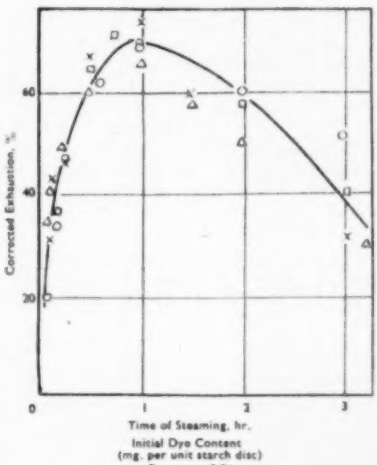


FIG. 5.—Transfer of Coomassie Brilliant Blue FF pure to Cellulose during Steaming at Different Initial Concentrations

per disc, suggesting that this amount of dye is less mobile and less fully available for transfer than any further amount of dye which might be present initially in the starch. It may be the result of a strong affinity between a small number of groups in the starch and the dye molecules. If now the exhaustion of this dye is calculated as the percentage of mobile dye available, and not of the total dye, in each case —

$$\text{Exhaustion} = \frac{\text{Dye in cellulose (mg.)} \times 100}{\text{Total dye (mg.)} - 0{\cdot}085}$$

and these exhaustions are all plotted against time of steaming, the graph in Fig. 5 results. All the points, regardless of the initial concentration of dye in the starch, group themselves round a single curve with a maximum corresponding to 1 hr. steaming time. There is some scatter, particularly for the longer steaming times, but the correction of the value for exhaustion has made this quantity much less dependent upon the quantity of dye in the system. This suggests that the transfer behaviour may be less a function of the intrinsic diffusion properties of the dye than of some constant property of the starch–cellulose system.

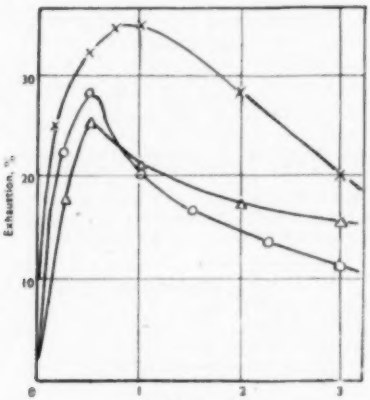


FIG. 6—Transfer of Coomassie Brilliant Blue FF pure to Cellulose during Steaming in presence of Assistants

Fig. 6 shows that the effect of additions of, respectively, sodium chloride and urea is very similar to that produced by the same substances on the transfer of Naphthalene Scarlet R, particularly in bringing about a lower general exhaustion and in causing a maximum in the steaming curve which is more sharply defined and which occurs earlier than when they are absent. Additions of small amounts of glycerol (0·7 ml. per 100 g. of dye paste) and of ammonia (15 ml. of 0·88 sp. gr. solution per 100 g. of paste) also cause a general decrease in the exhaustion and bring forward the time at which a maximum occurs.

It was found that a preliminary mercerisation of the cellulose film, by immersion for 3 min. in caustic soda (240 g./litre) followed by thorough washing in water, had comparatively little effect on the amount of dye transferred in steaming, except that transfer was more rapid in the early stages, so that the maximum occurred in about 30 min. instead of 1 hr. Steaming of the cellulose film for 21 hr. before use in a transfer experiment caused less than 2% change in the exhaustion after 1 hr. steaming.

As already noted, it was impossible to produce suitable films of dried starch if the urea content were too high. For investigating the effect of higher proportions of this reagent, therefore, the device was adopted of drying the dye-containing paste on the cellulose at room temperature before steaming (p. 79). During the process of drying there is considerable migration of dye to the cellulose. The results of preliminary experiments without the addition of urea are shown in Fig. 7.

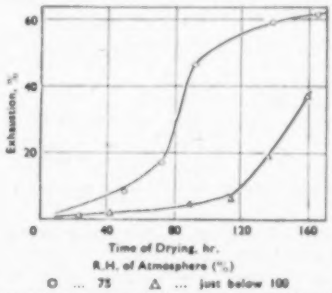


FIG. 7—Transfer of Coomassie Brilliant Blue FF pure from Layer of Starch Paste to Adjacent Film of Cellulose during Drying of Paste at Room Temperature

It will be seen that migration of dye does not depend merely upon time of contact between dye paste and cellulose, but progresses as water is evaporated from the starch. Drying into an atmosphere of 75% R.H. gives a relatively quick transfer. Slower drying into an atmosphere just below saturation with water vapour gives rise to a correspondingly slow transfer of dye. In each case, transfer is initially slow until a certain amount of the water is removed, and then increases rapidly. At the other end of the range, where most of the water in both starch and cellulose has been removed, the transfer, as might be expected, again becomes slow.

Following this, the experiments represented by Fig. 8 were carried out. Pastes containing, respectively, 3·5% and 9·7% of urea by weight were each spread to the standard thickness on cellulose and dried at room temperature as already described. Pieces of the composite films were then steamed, and the migration of dye was determined. It will be seen that the dye which had passed into the cellulose during the drying returns in large measure to the starch on steaming. The more urea that is present, the less dye is transferred to cellulose during drying or to starch during steaming. As an

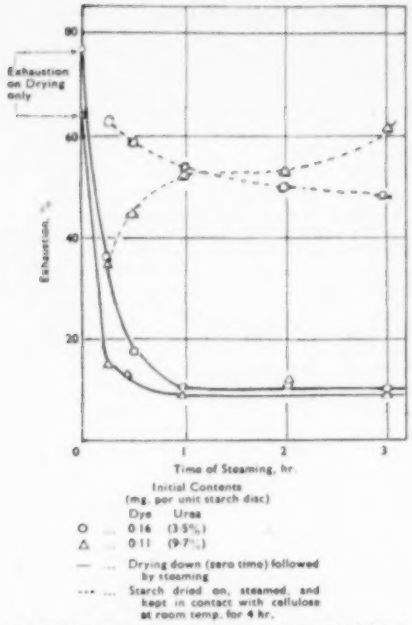

Exhaustion on Drying only
Exhaustion, %
Time of Steaming, hr.
Initial Contents (mg. per unit starch disc)
Dye Urea
O ... 0·16 (3·5%)
△ ... 0·11 (9·7%)
— ... Drying down (zero time) followed by steaming
--- ... Starch dried on, steamed, and kept in contact with cellulose at room temp. for 4 hr.

FIG. 8.—Transfer of Coomassie Brilliant Blue FF pure to Cellulose on drying down from Paste and then Steaming in presence of Urea

extension of this experiment, some of the combined starch–cellulose strips after steaming were allowed to stand, without separation, at room temperature for 48 hr. During this period, some of the dye which had returned to the starch during steaming again reversed its direction of migration and went finally to the cellulose.

COOMASSIE BRILLIANT BLUE R

The transfer of this dye without assistants is shown in Fig. 9. Its behaviour is distinguished from that of the two preceding dyes in that (*a*) there is no evidence of the formation of a maximum on the steaming curve, and (*b*) over the range of dye concentration examined there does not appear to be any simple relation between exhaustion and initial concentration of dye in the starch.

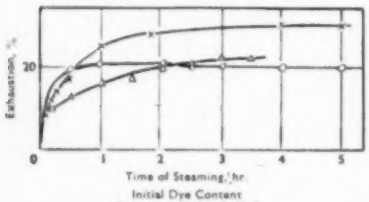


FIG. 9.—Transfer of Coomassie Brilliant Blue R pure to Cellulose during Steaming

The behaviour is, in fact, strongly reminiscent of that observed with Chlorazol Sky Blue FF[1]. As compared with Coomassie Brilliant Blue FF, the exhaustion is low. There is qualitative evidence that Brilliant Blue R, either through greater particle size or because of stronger chemical attachment between molecules of dye and thickener, cannot diffuse out of the starch film so readily as Brilliant Blue FF. If pieces of dried starch film containing one or the other of these dyes are dropped into cold water, there is immediate and heavy bleeding of Blue FF, while very little bleeding of Blue R occurs until the starch itself begins to disperse.

Addition of a small proportion of urea, equal in weight to the dye which is present, speeds up the migration of this dye to cellulose in the early stages of steaming (Fig. 10). A large proportion

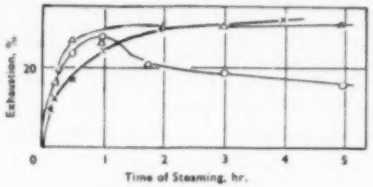


FIG. 10.—Transfer of Coomassie Brilliant Blue R pure to Cellulose on Steaming in presence of Urea

(ten times the weight of dye) causes the formation of a maximum in the steaming curve similar to that found with the two preceding dyes.

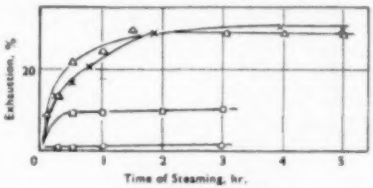


FIG. 14.—Transfer of Coomassie Brilliant Blue R pure to Cellulose in presence of Sodium Chloride

That the relatively slow migration of this dye is due rather to large particle size than to chemical attachment between molecules of dye on the one hand and molecules of thickener or fibre substance on the other seems to be indicated by the marked effect of sodium chloride in diminishing exhaustion (Fig. 11).

CARBOLAN YELLOW 3G

The solubility of this dye in water in the cold is not very great, and Fig. 12 shows that the transfer

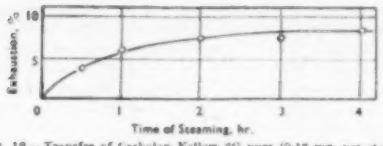
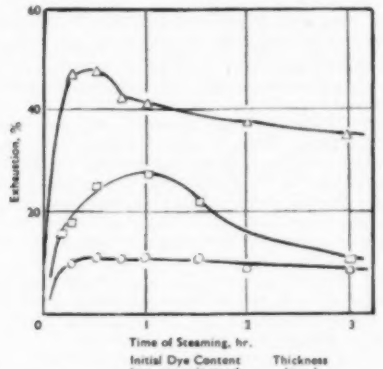


FIG. 12— Transfer of Carbolan Yellow 3G pure (0·16 mg. per starch disc) to Cellulose during Steaming

from starch to cellulose on steaming is small. The form of the exhaustion curve resembles that of Coomassie Brilliant Blue R. The behaviour of this dye was not investigated further in great detail, but the following observation tends to confirm Meitner's suggestion that the low exhaustion is due to large particle size, and that the implied strong tendency towards molecular aggregation will tend to cause any dye which can enter the cellulose to be firmly fixed. If a starch film containing the dye is attached to cellulose film and, instead of steaming, is maintained in the vapour above boiling pyridine for 1 hr., the transfer of dye to the cellulose is considerable, and practically no bleeding takes place if the cellulose is then boiled in water for 2 hr.

INFLUENCE OF RELATIVE THICKNESSES OF STARCH AND CELLULOSE FILMS

Most of the experiments were performed with Coomassie Brilliant Blue FF, and Fig. 13 gives



	Initial Dye Content (mg. per unit starch disc)	Thickness (mm.)
△ ...	0·15	0·06
□ ...	0·14	0·10
○ ...	0·14	0·23

FIG. 13— Transfer of Coomassie Brilliant Blue FF pure to Cellulose from Starch Films of Various Thicknesses

examples of the kind of results obtained. By spreading layers of starch paste containing dye to different thicknesses on glass and drying in the usual way, solid films of 0·23, 0·10, and 0·06 mm. were made. By adjustment of the weight of dye added to the paste, it was arranged that the weight of dye per unit area of film was the same for all thicknesses. It amounted to 0·14–0·15 mg. per disc ⅜ in. in diameter. The volume concentration of dye in this series of films was thus approximately in inverse ratio to the thickness. It will be seen that transfer from the thinner and more concentrated films is greater on steaming than from the thicker, and the maximum in the steaming curve is more pronounced.

An experiment in which the starch : cellulose ratio in the system is varied in the opposite direction, and which, incidentally, demonstrates the mobility of the dye within cellulose during steaming, was carried out with Naphthalene Scarlet R. Here, two strips of soaked Cellophane were squeegeed together before the dyed starch disc was attached to one side of the double strip, and the whole was steamed together. The results are shown in Fig. 14, in which are four curves,

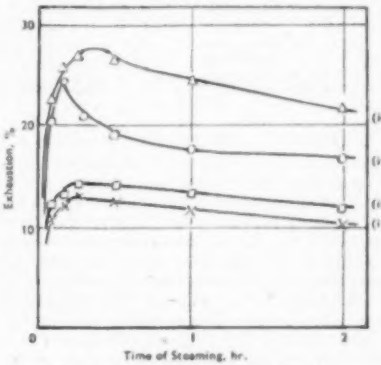
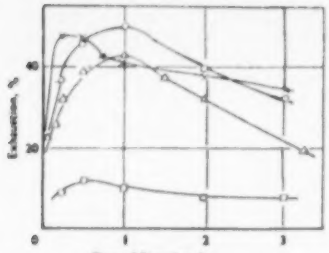


FIG. 14— Transfer of Naphthalene Scarlet R pure to Double Layer of Cellulose during Steaming (initial dye content 0·28 mg. per unit starch disc)

representing— (i) exhaustion of dye on the cellulose contiguous to the starch, (ii) exhaustion of dye on the cellulose sheet not in direct contact with the starch but separated from it by (i), (iii) the sum of the transfers in (i) and (ii), and (iv) the transfer to a single thickness of Cellophane in the ordinary manner. Comparison of (iv) with (iii) shows that an increase in the proportion of cellulose in the system brings about an increase in the proportion of the total dye which migrates from the starch, and that passage of dye from starch through the intermediate layer of cellulose to the outer one must be very rapid. These results, taken in conjunction with those for Coomassie Brilliant Blue FF in Fig. 3 and 13, suggest that, while the distribution of dye in the system during steaming is clearly related to the relative proportions of starch and cellulose, the formation of a characteristic peak in the exhaustion curve seems to be connected with the absolute concentration of dye in the cellulose phase.

Qualitative observations for similar systems in which the dyes Coomassie Brilliant Blue FF and Coomassie Brilliant Blue R were present show that these dyes also are capable of diffusing very rapidly through cellulose.



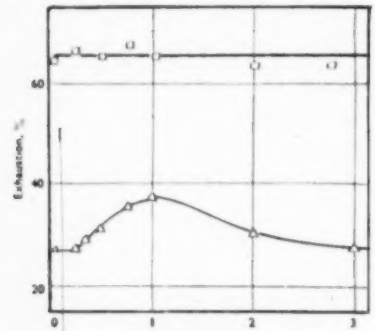
	Thickening	Initial Dye Content (mg. per unit disc)	Thickness (mm.)
×	Potato starch	0·15	0·05
O	Tragacanth	0·16	0·046
△	Potato starch	0·23	0·10
□	Colloresin DK	0·22	0·12

FIG. 15—Transfer of Coomassie Brilliant Blue FF pure to Cellulose from Potato Starch, Gum Tragacanth, and Methyl Cellulose respectively

EFFECT OF THE THICKENING AGENT

Here also, the trials were made with Coomassie Brilliant Blue FF. Reilly and Turner [5], in corresponding work with the direct dyes, have shown that it is often difficult to make comparisons between dyed films of different thickeners on the basis of a standard film thickness, since the mechanical properties of the different materials vary so widely. For this reason, with Colloresin DK and with gum tragacanth, a starch film matching each in respect of thickness and dye content was made for comparison. From Fig. 15 it will be seen that, while the behaviour with tragacanth is not very different from that with starch, except that at this film thickness the initial rate of transfer is lower, the migration from methyl cellulose is very poor.

There were many difficulties in preparing films of sodium alginate containing acid dyes and in attaching them to cellulose. With this thickener, therefore, the procedure already described was adopted in which the paste is spread directly on the cellulose film and dried at room temperature before steaming. From Fig. 16 it is seen that, in agreement with the results of Reilly and Turner for Chlorazol Sky Blue FF, the transfer of Coomassie Brilliant Blue FF at room temperature is much greater than from starch, and further, that the dye so transferred is retained completely by the cellulose during prolonged steaming.



	Thickening	Initial Dye Content (mg. per unit disc)
△	Potato starch	0·17
□	Sodium alginate	0·13

FIG. 16—Transfer of Coomassie Brilliant Blue FF pure to Cellulose from Starch and from Sodium Alginate, dried on at Room Temperature and Steamed

Discussion

The behaviour of the acid dyes, which requires special consideration, and for which a unifying explanation is sought, may be summarised as follows—

(i) The influence of dye concentration in the external dyebath (or thickener) upon the rate and extent of dye migration to the fibre substance.

(ii) Specifically, the evidence of reverse migration, i.e. dye transfer from the fibre substance to the thickener during the later stages of the steaming operation.

(iii) The effect on (i) and (ii) above of various auxiliary substances, particularly urea.

It should be made clear at this stage that the present work has been principally concerned with the migration of dye between thickener and fibre substance. Very little direct attention has yet been given to dye fixation, i.e. to the fastness of the dye in the cellulose of finished dyeings or prints towards wet treatments.

1. DYE CONCENTRATION AND DYE TRANSFER

The concentration at which the selected acid dyes are applied initially to the fibre substance is shown to be important for the following reasons among others—

(*a*) If these dyes are applied to cellulose fibres from solutions of the low concentrations customary in dyeing, only a very small proportion of the dye is taken up by the fibre. When the same weight of dye per unit weight of cellulose is applied at high concentration from the restricted volume of a film of thickener during drying and steaming of a print, much more dye is taken up.

(*b*) Transfer of dye to the fibre substance from an applied layer of starch paste increases progressively as the paste is dried and the dye in it becomes more concentrated.

(*c*) The proportion of the total dye migrating during steaming from a film of starch to a film of cellulose in contact with it increases with the initial concentration of dye in the starch.

(*d*) In film-to-film systems with the same weight of dye per unit area of contact, the proportion of dye migrating to the starch during a fixed period of steaming increases as the starch film is made thinner or as the cellulose film is made thicker.

The first approach towards the solution of these problems can be made by considering a simple system in which contiguous layers of water-swollen starch and water-swollen cellulose remain constant in volume and in water content, the dye being molecularly dispersed and the molecules being free to diffuse in the water of each phase, subject to chemical attraction neither from cellulose nor

from thickener molecules. Initially, the dye is all in the thickener phase, and it will diffuse under the influence of a concentration gradient into the cellulose until the concentration of dye in water-in-cellulose is the same as that in water-in-starch, when an equilibrium is established. The time required to set up equilibrium in this simple system will vary from dye to dye according to the diffusion coefficient in water, but the ultimate distribution between the two phases of any dye which can be conceived to fulfil the required conditions will be the same. This distribution will be determined only by the ratio in which the total water content of the system is divided between the two phases. Extending this idea, and assuming that the inter-phase area and total weight of dye in the system remain constant, it will be seen that any influence which alters the water content of one phase relative to the other will bring about a corresponding change in the equilibrium distribution of the dye. Thus, decreasing the thickness of the starch phase by drying will increase the proportion of total dye in the cellulose. Altering the relative weights of fibre substance and thickener, e.g. increasing the thickness of one film while the other remains constant, will cause more dye to be retained after steaming in the film whose thickness has been increased. Addition of swelling agents which cause one phase to take up a relatively greater proportion of water than the other on steaming, or to retain more water on drying, will cause an increased retention of dye in that film.

2. CAUSE OF MAXIMUM

Many aspects of the behaviour of actual systems as described in the earlier part of this paper are consistent with the simple mechanism just propounded, but, because it is greatly simplified, it will obviously not be able to explain all the observed properties either of the experimental systems or of an industrially produced print. Thus, it does not explain the maximum which is observed on the steaming curves of some of the dyes. This maximum is caused quite definitely by the reverse transfer of dye from cellulose to starch during the later stages of steaming, and is not due to destruction of colouring matter by heat and the possible reducing action of thickener and cellulose.

One obvious departure from the postulated ideal conditions is that the water contents of the two phases are unlikely to remain constant or to preserve a constant ratio one to another during the course of steaming in an actual experiment. Work in this laboratory by G. D. Myers and R. E. Pomfret has shown that there are very serious difficulties in following the changes in water distribution between the two phases when a starch film is steamed in contact with a cellulose film. Present evidence shows that the water content of an isolated film of soaked cellulose falls very rapidly during the first few minutes after introduction into steam and then remains practically constant. On the other hand, the water content of a dry starch film in steam rises at first rapidly, and then slowly and continuously for at least 5 hr. This does not represent the complete possibility of swelling, since it is found that prolonged steaming, corresponding to 7 hr. or more, causes the starch to run. While this is doubtless caused by decomposition of the starch, with a consequent lowering of viscosity, the loss of adhesion between one thickener molecule and another is likely to bring about a further uptake of water. No reliable results have been obtained for this stage.

While further and more precise data on this point are being sought, the following hypothesis appears to be the most useful and the most consistent with the available evidence. When a dry film of starch containing acid dye is squeegeed into contact with a water-soaked film of cellulose, water will first pass rapidly into the starch from the cellulose. On putting into steam, this adjustment of water content between the two phases will continue for a short time. At the same time, as the starch film is opened up, dye, at high concentration in the water contained in the film, will diffuse into the cellulose, tending towards an equalisation of dye concentration in the water of the two phases. In the meantime, more slowly, preferential acquisition of more water by the starch and dilution of the dye therein will slow down and ultimately reverse the diffusion from this phase to the cellulose. If the speed of dye diffusion, as steaming proceeds, is suitably related to the differential swelling just postulated, the formation of a maximum in the exhaustion curve soon after the beginning of the experiment is easy to explain.

3. FURTHER DEVELOPMENT OF DISTRIBUTION HYPOTHESIS

In enunciating the simple hypothesis, no account has been taken of the chemical attraction of the dye for either the thickener or the fibre molecules. Although, with most of the dyes examined, it does not appear to be large for cellulose, since the cellulose cannot adsorb much dye from dilute solutions, it is unlikely to be zero. The same is true as far as starch is concerned. The simple hypothesis requires that when standard starch and cellulose films are combined and steamed by the same procedure, the relative water contents of each will always be the same at the end of a given time. If the diffusion of the dye is sufficiently rapid to allow equilibrium concentration in the water of the system to be approached before the movement of water from the cellulose, due to preferential swelling of the starch, becomes appreciable, this should mean that dye also will always be distributed between the two phases in the same ratio at the end of the same steaming time, when this time is not too short. This ratio would be independent of the actual amount of dye in the system.

Fig. 4 indicates that this condition is approached for Coomassie Brilliant Blue FF. If the constant distribution hypothesis were true, the straight line when produced should pass through the origin. In fact, it cuts the dye-in-starch axis at a value of 0·085 mg. per disc. At concentrations below this value, it would seem that either (*a*) the initial concentration gradient in the system is so small that the rate of forward diffusion into the cellulose cannot keep pace with the uptake of water by the starch, so that the dye is, so to speak, sucked back

into the expanding thickener phase, or (*b*) there are a few active centres in the starch (e.g. ionic groups) which can bind a certain proportion of dye much more strongly than the rest. The alternative (*a*) is the less attractive. The exhaustions at 1 hr., while small, are not negligible for initial concentrations of 0·06 and 0·04 mg. per disc, and, on the other hand, it is difficult to see why the effect should stop suddenly at 0·085 mg. per disc, rather than fade out slowly as higher initial concentrations are used. Protein impurity in the starch could be responsible if explanation (*b*) is accepted. It will be seen that the value of 0·085 mg. per disc forms a dividing line in the behaviour of the systems. Below this initial dye concentration, there is no sign of a maximum in the exhaustion curve. A re-examination of analogous systems by Pomfret has shown that this is not to be ascribed to the relatively greater effect of experimental errors at these low concentrations. The curves below this concentration resemble those for Coomassie Brilliant Blue R; above it they show the characteristic maximum. The former curves therefore seem to show the type of migration to be expected in systems where all the dye is under constraint by the thickener, the latter where some of the dye is much more free to diffuse.

The hypothesis of diffusion primarily to cellulose, opposed by preferential swelling of starch, may thus appear to explain the presence of a maximum in the exhaustion curve of Naphthalene Scarlet R or Coomassie Brilliant Blue FF when either dye is considered separately, but lack of precise information about the swelling behaviour of the system and the distribution of water in the two phases makes it difficult to interpret differences in behaviour of the two dyes. With Naphthalene Scarlet R (and also with Naphthalene Red EA) the maximum exhaustion occurs within 10 min. after the commencement of steaming; with Coomassie Brilliant Blue FF, 60 min. are needed. On Meitner's evidence, the particle size of the latter is much the greater, and therefore the specific rate of diffusion is correspondingly lower. How far the difference in particle radius as between one dye and the other will be preserved at 100°C. is not yet known, but the general indications remain that, for one reason or another, the Scarlet R can diffuse more quickly than the Brilliant Blue FF. From Fig. 1 it appears that the principal migration of dye to the cellulose occurs very quickly with Scarlet R, and that very soon afterwards this migration is opposed and then reversed by the preferential swelling in the starch, which is substantially completed (on the evidence of the exhaustion curve) in about 40 min. Turning to Fig. 3 for Coomassie Brilliant Blue FF, it is now seen that the initial forward migration of dye is predominant for at least 40 min., and the preponderating effect of a factor causing reverse migration does not set in for about 50 min., and then continues for 180 min. and longer. It seems impossible that the reverse migration of Scarlet R should be caused by the same changes in the starch–cellulose system as those which cause the reverse migration of Brilliant Blue FF. Nor can a way out of the discrepancy (an almost similar one has been observed by Pomfret in comparing the transfer behaviour of Naphthalene Red EA with that of Coomassie Violet 2R) be found merely by assuming that the preferential water uptake of starch continues in stages throughout a prolonged steaming, Scarlet R being affected by an early stage, Brilliant Blue FF "missing" this on account of its more sluggish movement and being affected by a later one. If this were true, it would be expected that the exhaustion curve of Scarlet R would show a further drop as steaming continues up to, say, 1 hr. and beyond. A drop of this kind is not observed. Further attempts to solve this problem by a differential swelling hypothesis must be regarded as very speculative at present. One of these is to assume that a dye like Brilliant Blue FF, which is rather large and sprawling in its molecular configuration, can, in its aggregated form, exercise a kind of cementing action on starch molecules, opposing swelling in the starch film until some of the dye has broken free and migrated to the cellulose. It would be difficult on this basis to explain the observed fact that the maximum exhaustion always occurs at the same time, although the dye content of the system may vary over a wide range.

4. EFFECT OF CHANGES IN AGGREGATION OF DYE MOLECULES

Further examination may ultimately prove that the formal similarities in the migration behaviour of dyes such as Naphthalene Scarlet R and Coomassie Brilliant Blue FF are brought about by quite different causes. The migration characteristics of Naphthalene Scarlet R may in fact be an example of the working of a mechanism such as we have discussed. The migration of dyes like Coomassie Brilliant Blue FF, with larger particle radius, may prove to be controlled largely by changes in the state of aggregation of the dye as envisaged by Meitner. With the limited amount of water available in the starch phase, the aqueous dispersion of the dye will be concentrated, and the dye will therefore be encouraged to form large particles. These must be made to break down under the influence of the steaming conditions into much smaller units before they can pass through the starch film and enter into the cellulose. This breakdown may be a relatively slow process as compared with the diffusion, and so the total migration rate will be slow as compared with that of a dye with less tendency towards aggregation. On Meitner's theory, it is assumed that re-aggregation of small particles to large ones will follow the entry of dye into the cellulose, and, if the system is truly polydisperse, will lead to further entry of smaller particles into the cellulose until an equilibrium is established. Extending this idea, it is necessary to find some change in the system which will, after, say, 40 min., cause the large aggregates of dye in the cellulose to redisperse and to migrate back to the starch. This seems a very obvious field for enquiry. It may be that the change is initiated by increased swelling of the starch, which we have invoked in the simpler theory. If the processes of aggregation and disaggregation are relatively slow, they will impose a time lag

between the initial swelling in the starch and the migration away from the cellulose which, it has been suggested, is the ultimate result of this swelling. Some support is found for this idea in the migration behaviour of Coomassie Brilliant Blue FF in the presence of urea. Fig. 6 shows that addition of a small proportion of urea lowers the value for maximum exhaustion and brings forward the time at which it occurs. Urea may be regarded both as a swelling agent for starch or cellulose and as a dispersing agent for the dye. It is to be expected that the loosely bound structure in the starch film will be more readily swollen than the more strongly bound one in the cellulose. Thus, in a given steaming time, the increased swelling of the starch will lead to a diminished exhaustion of dye. At the same time, the dispersing action of the urea on the dye particles will cut down the time lag discussed above and cause the dye to follow more quickly the swelling changes in the starch–cellulose system. With Coomassie Brilliant Blue R, it may be assumed that the rate of dispersion of dye particles originally in the starch phase into the smaller ones capable of migrating into the cellulose is so slow that transfer to the cellulose is almost completely uninfluenced by the swelling changes in the system. Only when the dye is further dispersed by a substantial proportion of urea is the migration quick enough to be able to reflect the swelling changes.

Further support is given by the transfer curves of Carbolan Yellow 3G (Fig. 12) and of Naphthalene Scarlet R in the presence of Fixanol (Fig. 2) for the suggestion that if the aggregation of the dye molecules is very pronounced, and the rate of diffusion correspondingly diminished, the differential swelling of the system will be without effect.

So far, a detailed examination of the diffusion of the acid dyes within the layers of starch and of cellulose respectively, by the cutting of cross-sections and viewing with the microscope, has not been attempted. The mobility of some of these dyes does introduce certain difficulties in the preparation of representative sections, but it is hoped to initiate work of this kind. By so doing it may be possible to decide upon the validity of an alternative proposal to explain the presence of a maximum in the exhaustion curves of some of the dyes. In this it is assumed that, for some reason not clearly understood, rapid transfer of dye from the starch to a thin adjacent layer of the cellulose takes place during the early stages of steaming. The dye is supposed to aggregate in this layer without much diffusion into the body of the cellulose, so that a very high local concentration results. In popular language there is a heavy strike on the cellulose. Further steaming can lead to a breakdown of the aggregates of dye in the concentrated layer with the possibility of diffusion in both directions, viz. further into the cellulose and back into the starch. This proposal is similar to a suggestion made by Lemin and Rattee [6] to explain certain peculiarities of the dyeing curves of levelling acid dyes on wool.

* * *

We should like to express our sincere thanks to Dr. S. H. Mhatre, now of the University of Bombay, for many valuable suggestions and discussions during the course of the work. Thanks are due also to Imperial Chemical Industries Ltd., who furnished the dyes used in the investigation, to the Manchester Education Committee for a scholarship, and to Sheth Padambhai Harida for other financial help which enabled one of us (R.B.P.) to take part in the work.

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The Action of β-Propiolactone on Wool

C. Fearnley and J. B. Speakman

There is little reaction between dry wool and β-propiolactone when the latter is dissolved in anhydrous benzene, but reaction is vigorous in alcoholic solution at 50°c. Combination is not confined to the basic side-chains of salt linkages, though 50% of the latter are broken during 48 hours' treatment. As the resistance of the fibres to deformation in water and acid is reduced, it was expected that treated fabric would show an increased rate of shrinkage during milling, especially as the percentage hysteresis between extension and contraction is reduced. Little difference could, however, be detected between treated and untreated fabrics during milling with soap or acid, and the felting power of loose wool is reduced by treatment with the lactone, probably because the beneficial effects of modified elastic properties are offset by reduced scaliness of the fibres.

A high proportion of the side-chains of keratin carry free amino, carboxyl, hydroxyl, and phenolic groups, which are known to react with β-propiolactone in the following manner [1]—

$$\begin{array}{l} R\cdot NH_2 + \begin{array}{l} CH_2\cdot CH_2 \\ | \quad\quad | \\ O\!-\!-C{:}O \end{array} \rightarrow R\cdot NH\cdot CO\cdot CH_2\cdot CH_2OH \\ \\ R\cdot XH + \begin{array}{l} CH_2\cdot CH_2 \\ | \quad\quad | \\ O\!-\!-C{:}O \end{array} \rightarrow R\cdot X\cdot CH_2\cdot CH_2\cdot COOH \end{array}$$

(XH = COOH or OH). When wool is treated with the reagent, therefore, its acidity should be increased, and the breakdown of salt linkages, when the basic side-chains are converted into hydroxylic and, ultimately, acid side-chains, should increase the ease of deformation of the fibres in water. In the absence of complicating factors this, in turn, should give the increased felting power which has been reported by Jones and Lundgren [2]. They recommend that the wool should be dried, and then treated at 40°c. with at least 30% by weight of the lactone in a dry solvent, such as ether or benzene, until the gain in weight is sufficient to reduce the nitrogen content of the product to 12%. In the light of these observations it seemed probable that di-β-lactones would be specially effective cross-linking agents for wool, and the following experiments with β-propiolactone were carried out to obtain further information about its action on the fibre, as a basis for subsequent work on cross-linking with di-β-lactones.

Experimental

Preparation of Materials

(*a*) Lincoln Wool—After cutting off the exposed tips, the root ends of the staples were purified by extraction with alcohol and ether in a Soxhlet apparatus, followed by washing in distilled water.

(*b*) Flannel—Square patterns weighing 2·5 g. were cut from an all-wool flannel having the following characteristics—

Warp	...	30s Yorkshire skeins with 10·4 t.p.in.; 35 ends/in.
Weft	...	30s Yorkshire skeins with 10·4 t.p.in.; 33 picks/in.
Weave	...	Plain
Weight	...	5·5 oz./sq. yd.

The patterns were purified in the same way as the Lincoln wool.

(*c*) Hosiery Fabric—Square patterns weighing 8–9 g. were cut from an all-wool knitted fabric having the following characteristics—

Yarn	...	1/20·4s worsted, knitted double
Wales/in.	...	18
Courses/in.	...	24
Weight	...	9·7 oz./sq. yd.

The patterns were purified in the same way as the Lincoln wool.

(*d*) β-Propiolactone—The purity of the commercial product was estimated by titration with sodium hydroxide and found to be 98·2%.

Preliminary Experiments

In these experiments dry flannel was treated with solutions of β-propiolactone in anhydrous solvents, as recommended by Jones and Lundgren [2]. Each sample was dried in an air-oven at 104°c. for 3 hr., and weighed after being allowed to cool over phosphorus pentoxide *in vacuo*. The pattern was then immersed in 100 c.c. of a solution containing 1·25 c.c. of β-propiolactone, dissolved in anhydrous ether or benzene, for 48 hr. at 25°c. or 50°c. Anhydrous ether was prepared by drying the commercial product over sodium wire, and then fractionally distilling in a stream of dry air, the distillate being stored over sodium wire. The benzene was also dried over sodium wire. After treatment, the flannel was rinsed with the organic solvent, and then washed in running water overnight before its dry weight was determined in the same manner as before. The gains in weight of the patterns, which are given in Table I, are far smaller than the 12% reported by Jones and Lundgren.

That the lactone should fail to react to any significant extent with the flannel is, no doubt, due

Table I

Treatment		Increase in Weight (%)
β-Propiolactone in ether at 25°c.	...	0·95
		0·98
β-Propiolactone in benzene at 50°c.	...	1·25
		1·24

to perfect dehydration of both the wool and the solvents. The fine structure of dry fibres is difficult of access to molecules as small as those of *n*-propyl alcohol [3] and, in addition, traces of water may be essential to the reactions with keratin, because it has been shown that thiourea [4] does not react with β-propiolactone in non-polar solvents, although reaction does occur in water. Despite the probability of loss of reagent by reaction with the solvent, further experiments were carried out with solutions of β-propiolactone in absolute alcohol, which swells the fibres, using wool from which traces of water were not excluded.

REACTION BETWEEN WOOL AND ALCOHOLIC SOLUTIONS OF β-PROPIOLACTONE

After the samples of flannel had been dried and weighed, they were each transferred, without taking precautions to prevent adsorption of water vapour from the air, to 100 c.c. of a solution containing 1·25 c.c. of β-propiolactone. Treatment was carried out for 48 hr. at 25°c. or 50°c., and the observed increases in dry weight of the washed patterns are given in Table II. Included in the latter are values for the changes in weight of patterns which were treated with absolute alcohol alone under similar conditions.

TABLE II

Treatment	Increase in Weight (%)
β-Propiolactone at 25°c.	11·6
	13·6
Alcohol at 25°c.	− 1·1
	0·2
β-Propiolactone at 50°c.	21·3
	22·0
Alcohol at 50°c.	− 0·9
	0·2

Similar large increases in weight were obtained when purified samples of the knitted fabric were treated with alcoholic solutions of β-propiolactone for 48 hr. at 25°c. and 50°c. (Table III).

TABLE III

Treatment	Increase in Weight (%)
8·0-g. pattern in 100 c.c. solution containing 4·00 c.c. β-propiolactone at 25°c.	16·2
8·5-g. pattern in 100 c.c. alcohol at 25°c.	− 1·1
9·0-g. pattern in 100 c.c. of solution containing 4·50 c.c. β-propiolactone at 50°c.	25·2
8·0-g. pattern in 100 c.c. alcohol at 50°c.	− 1·0

PROPERTIES OF WOOL TREATED WITH ALCOHOLIC SOLUTIONS OF β-PROPIOLACTONE

(1) *Milling Shrinkage*

(*a*) SOAP MILLING—Flannel and hosiery patterns were treated with β-propiolactone under the above conditions and, after being washed in running water, were allowed to relax in warm soap solution for 30 min. before their areas were measured. The two sets of patterns were then milled by hand for 10 min., at the same time as an untreated pattern, using a 3% solution of soap as the milling agent. The resulting shrinkages are given in Table IV.

TABLE IV

Treatment	Shrinkage in Area (%)
FLANNEL	
Untreated	32·2
β-Propiolactone at 25°c.	34·9
Alcohol at 25°c.	34·1
β-Propiolactone at 50°c.	28·5
Alcohol at 50°c.	30·2
HOSIERY FABRIC	
Untreated	33·3
β-Propiolactone at 25°c.	31·8
Alcohol at 25°c.	28·6
β-Propiolactone at 50°c.	32·3
Alcohol at 50°c.	29·1

Treatment with β-propiolactone has surprisingly little effect on the milling shrinkage of the patterns, and similar results were obtained with the flannel when hydrochloric acid was used as the milling agent.

(*b*) ACID MILLING—After the patterns had been treated with an alcoholic solution of the reagent or alcohol alone, under the same conditions as before, 1% by weight of oleic acid was deposited on each from alcoholic solution in order to promote shrinkage during acid milling[4]. The patterns, with an untreated pattern to which 1% oleic acid had also been applied, were then immersed in three changes of 0·1 N. hydrochloric acid (final pH = 0·96) and, after their areas had been measured, they were milled by hand for 15 min. Rubber gloves were worn while milling was carried out, and the equilibrium solution of hydrochloric acid was used as the milling agent. The observed shrinkages are given in Table V.

TABLE V

Treatment	Shrinkage in Area (%)
Untreated	47·0
β-Propiolactone at 25°c.	40·5
Alcohol at 25°c.	39·7
β-Propiolactone at 50°c.	40·2
Alcohol at 50°c.	40·2

(2) *Felting*

In view of Jones and Lundgren's claim that wool treated with β-propiolactone has specially enhanced felting properties, it was surprising that treated flannel and hosiery fabric should fail to show increased shrinkage during milling. Unfortunately, Jones and Lundgren have not described their method of testing the treated wool, and it is possible that the "enhanced felting properties" do not imply an increased rate of felting but refer to the greater tensile strength of the felts, which is also claimed. It was, therefore, decided to determine the rate of felting of loose wool after treatment with β-propiolactone.

TABLE VI

Time of Felting (min.)	Compression Resistance of— Untreated Wool	Wool treated at 25°c.	Wool treated at 50°c.
10	0·543	0·573	0·477
15	0·666	0·614	0·512
20	0·689	0·633	0·564
25	0·707	0·671	0·588
30	0·737	0·689	0·611
40	0·762	0·737	0·623
50	0·758	0·735	0·636
60	0·789*	0·751	0·637
70	0·781	0·730	0·658

* This value was obtained at 62 min.

Samples of loose wool were treated with the reagent for 48 hr. at 25°c. or 50°c. under the usual conditions, the resulting gains in weight being

14·4 and 29·6% respectively. After the wools had been carded by hand, 1·7-g. samples were wetted with 1·7 c.c. of a 10% solution of sodium oleate and felted in the machine devised by Chamberlain and Menkart [5]. The "compression resistance" of the samples was measured at intervals (Table VI).

In the light of these results, there can be no doubt that the rate of felting of wool is reduced by treatment with β-propiolactone, but it should be noted that equal weights of treated and untreated wool were used, so that less keratin was present in the treated samples.

(3) *Elastic Properties*

In order to discover the cause of the unexpected behaviour of the treated wools during felting and milling, as well as to obtain further information about the nature of the reaction between keratin and β-propiolactone, the elastic properties of treated fibres were next examined. Lincoln fibres were calibrated by determining their load–extension curves up to 30% extension in distilled water and, after 24 hours' rest, in 0·1 N. hydrochloric acid, at 22·2°C. The fibres were then attached to 2·5-g. samples of flannel, treated with alcoholic solutions of β-propiolactone under the usual conditions, and finally washed in running water overnight. After the load–extension curves of the fibres had been redetermined in water and 0·1 N. hydrochloric acid at 22·2°C., values for the

TABLE VII

Treatment	Percentage Reduction in Resistance to Extension				
	Untreated Fibres in HCl (*B*)	Treated Fibres in Water (*C*)	Treated Fibres in HCl (*D*)	(*D* − *B*)	*C* − (*D* − *B*)
β-Propiolactone at 25°C.	35·3	22·9	45·6	10·3	12·6
	34·2	24·9	44·8	10·6	14·3
	34·9	24·4	43·5	8·6	15·8
Alcohol at 25°C.	38·5	− 0·3	37·5	− 1·0	0·7
	36·3	0·0	38·5	2·2	− 2·2
β-Propiolactone at 50°C.	36·0	31·7	49·5	13·5	18·2
	38·0	28·3	48·0	10·0	18·3
	38·7	30·2	50·4	11·7	18·5
Alcohol at 50°C.	—	2·7	40·4	—	—
	38·7	3·0	39·8	1·1	1·9

change in resistance to 30% extension, due to the various treatments, were derived from the several load–extension curves, and the results are summarised in Table VII. Each value is expressed as a percentage of the work required to stretch the original, untreated fibre to 30% extension in distilled water.

As the lactone-treated fibres are much less resistant to extension in 0·1 N. hydrochloric acid than the untreated fibres, as may be seen from the values of (*D* − *B*), it is evident that the reaction is not confined to the basic groups of salt linkages. The extent to which the latter are attacked is indicated by the values of *C* − (*D* − *B*), about 50% (18·3/37·6) being broken by reaction with the lactone at 50°C.

According to the theory which was advanced some years ago [6], milling shrinkage is promoted by increasing ease of deformation of the fibres, provided that their scaliness and power of recovery from deformation are unimpaired. Since the lactone-treated fibres are so much more easily deformed than untreated fibres in both water and hydrochloric acid, treated fabrics should shrink more rapidly than untreated during milling, but no such effect was observed. The cause of failure does not lie in a reduced power of recovery from deformation, because the percentage hysteresis [6] between extension and contraction in water is less for treated than for untreated fibres (Table VIII).

TABLE VIII

Treatment	Hysteresis (%)	
	Untreated Fibres	Treated Fibres
β-Propiolactone at 25°C. ...	55·6	50·4
	53·4	45·3
	52·9	48·6
Alcohol at 25°C.	56·2	55·7
β-Propiolactone at 50°C. ...	56·1	50·7
	58·9	49·3
	57·9	51·0
Alcohol at 50°C.	57·7	57·5
	56·9	57·2

In the light of these results, it seemed probable that the failure of the lactone-treated fibres to show an increased power of felting and milling is due to reduced scaliness.

(4) *Scaliness*

After their coefficients of friction (μ_1 and μ_2) had been measured by the capstan method [7] in a 0·5% solution of soap at 22·2°C., groups of Lincoln fibres were treated with alcoholic solutions of β-propiolactone in presence of 2·5-g. samples of flannel under the usual conditions. The fibres were washed in running water overnight before their coefficients of friction (Table IX) were redetermined.

TABLE IX

Coefficients of Friction

Treatment	Untreated Fibres			Treated Fibres		
	μ_1	μ_2	$\frac{\mu_1-\mu_2}{\mu_1+\mu_2}$	μ_1	μ_2	$\frac{\mu_1-\mu_2}{\mu_1+\mu_2}$
β-Propiolactone at 25°C.	0·44	0·27	0·24	0·40	0·27	0·19
	0·44	0·29	0·21	0·40	0·31	0·13
Alcohol at 25°C.	0·43	0·32	0·15	0·44	0·33	0·14
	0·47	0·28	0·25	0·48	0·29	0·25
β-Propiolactone at 50°C.	0·43	0·26	0·25	0·36	0·32	0·06
	0·48	0·25	0·32	0·45	0·31	0·18
	0·43	0·32	0·15	0·40	0·35	0·07
Alcohol at 50°C.	0·46	0·28	0·24	0·46	0·29	0·23
	0·45	0·22	0·34	0·45	0·24	0·30

In all cases, the scaliness of the fibres is reduced by treatment with β-propiolactone.

Discussion

The increased ease of deformation of wool fibres in water and 0·1 N. hydrochloric acid, after treatment with an alcoholic solution of β-propiolactone at 25°C. or 50°C., is not due solely to combination of the reagent with the basic side-chains of salt linkages. Cohesion due to van der Waals forces and hydrogen bonds must also be reduced by the swelling which takes place[2] when bulky side-chains are formed by combination of amino, carboxyl, hydroxyl, and phenolic groups with one or more molecules of the lactone. As the increased ease of deformation is accompanied by reduced hysteresis between extension and contraction, the felting and milling power of the treated wool should be greater than that of untreated wool. The fact that the felting power of wool is reduced by treatment with β-propiolactone must, therefore, be attributed to the reduced scaliness of the fibres. It is, however, probable that increased felting would be obtained if reaction with the lactone were confined to the tip halves of the staples, though the same result could be obtained more simply by means of reagents such as sodium bisulphite[6].

* * *

The authors are indebted to the International Wool Secretariat for grants in aid of this investigation.

TEXTILE CHEMISTRY LABORATORY
DEPARTMENT OF TEXTILE INDUSTRIES
THE UNIVERSITY
LEEDS

(Received 9th October 1951)

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ERRATA

Comparison of Fading Rates by means of the Society's Light Fastness Standards (B.S. 1006). N. F. Desai and C. H. Giles (J.S.D.C., **67**, 377 (Oct. 1951))— In Fig. 1 the ordinates ($a_0 - a$) should be -2, -1, 0, 1 instead of $-5 \ldots -2$.

Development of the Society's Washing Fastness Tests for Coloured Cellulosic Materials. L. A. Lantz (J.S.D.C., **67**, 442 (Nov. 1951))— In Table II the temperature of Tests No. 3 and 4 should be 185°F.

Notes

Proceedings of the Council

At a meeting of the Council, held at the Victoria Hotel, Bradford, on 9th January 1952, the proceedings included the following items of interest—

RIBBON FOR PRESIDENTIAL BADGE— It was reported that the Wardens of the Worshipful Company of Dyers considered the design and colour of the proposed new ribbon to be very appropriate. This ribbon would contain a ½ in. white band on 1 in. mauve.

A.A.T.C.C. ANNUAL CONVENTION— Mr. G. S. J. White reported that he had been warmly welcomed at the Thirtieth Annual Convention of the American Association of Textile Chemists and Colorists held in New York on 17–19th October 1951. He had presented specially bound copies of Volume I of the *Review of Textile Progress* and of the *Symposium on Photochemistry in relation to Textiles*, the latter including the coloured reproduction of the Presidential Badge; these small gifts had been greatly appreciated. He had been asked to convey the kindest regards and warmest greetings from the President and Council of the A.A.T.C.C. to the President and Council of the Society.

REPORT OF THE BYE-LAWS COMMITTEE— This report was adopted and the accompanying draft set of new Bye-laws was accepted, subject to certain amendments, for presentation to the Annual General Meeting of the Society in March 1952.

MEMBERSHIP — Thirteen applications for Ordinary Membership and seven for Junior Membership were approved. Mr. H. Hudson was readmitted to membership of the Society.

Meetings of Council and Committees February

Council— 13th
Finance— 13th
Publications— 19th
Colour Index Editorial Panel— 1st
Fastness Tests Co-ordinating— 22nd
Summer School— 6th and 23rd

Summer School 1952

Arrangements are in hand for a Summer School in Dyeing to be held at Loughborough College (Leicestershire) during 23rd–30th August 1952. A descriptive brochure is enclosed with this issue of the *Journal*, and also an application form, which should be completed and returned without delay by those who wish to attend the Summer School.

Printing Symposium 1953

Council has set up a small committee, consisting of Messrs. F. Farrington, N. Hamer, R. J. Hannay, J. W. Reidy, and G. S. J. White, to organise a Symposium on Printing, to be held during 1953.

Colour Index Second Edition Signing of Agreement

The formal agreement between the A.A.T.C.C. and the Society for the production of the Second Edition of the *Colour Index* was signed on 18th January 1952. On behalf of the Society, the agreement was signed by the President (Mr. F. Scholefield), the Honorary Treasurer (Mr. H. Jennison), and the Honorary Secretary (Mr. J. Barritt), in the presence of the United States Consul in Bradford. In New York the document received the seal of the A.A.T.C.C. and was signed by the President and the Secretary of the A.A.T.C.C.; the signing was acknowledged by a notary public, the County Clerk and Clerk of the Supreme Court, New York County, State of New York, and the British Consulate General, New York. The accompanying photograph shows the signing in New York.

Mr. W. A. HERRMANN
Attorney

Dr. E. R. LAUGHLIN
Secretary A.A.T.C.C. Colour Index Committee

Dr. H. C. CHAPIN
Secretary A.A.T.C.C.

Mr. C. N. RABOLD
President A.A.T.C.C.

Mr. W. H. CADY
Chairman and Co-ordinator A.A.T.C.C. Colour Index Committee

Manufacturers' Publications and Pattern Cards

The Society does not accept any responsibility with regard to the statements in the following notes. Any publication abstracted may be referred to by members of the Society on application to Dr. C. B. Stevens, Dyeing Department, Leeds University.

Badische Anilin- & Soda-Fabrik

INDANTHREN, ANTHRA, AND BRILLIANT INDIGO DYESTUFFS FOR PRINTS ON COTTON FABRICS.—This pattern card contains prints on cotton piece in two depths of shade of thirty-five Indanthren, eight Brilliant Indigo, and three Anthra vat dyes. The text, which is reproduced in English, German, French, and Spanish, is particularly detailed, and contains full particulars of available methods of printing, the fastness properties of the illustrated prints, and recommended uses of individual dyes.

British Celanese Ltd.

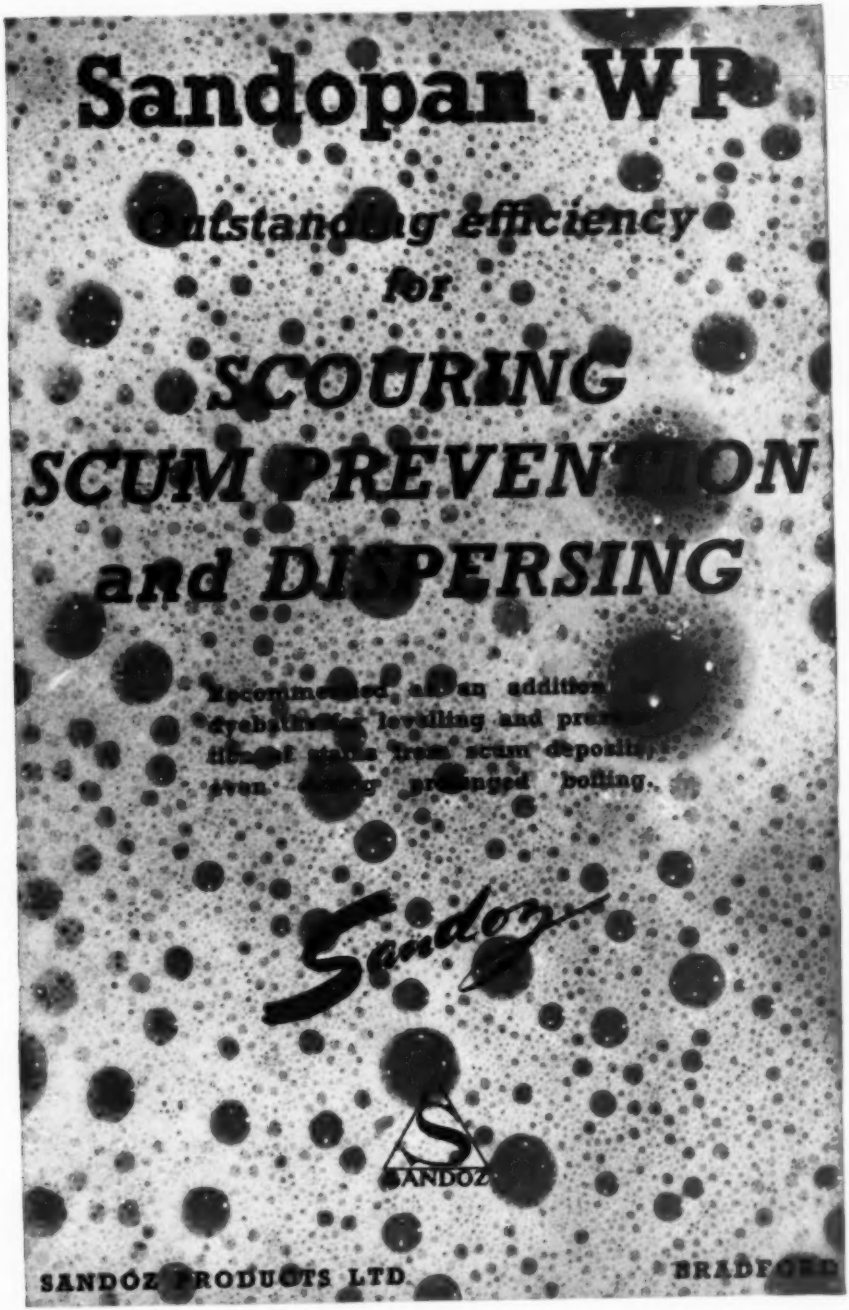
"CELANESE" S.R.A. DYESTUFFS.—This pattern card contains twenty-nine dyeings of individual disperse acetate rayon dyes of the S.R.A. series and three mixtures. Fastness data are included on separate sheets provided with the card of dyeings.

Compagnie Française des Matières Colorantes

SOLANTHRENE PRINTING BLACK BL PASTE OPTIMA.—This leaflet (in French) describes a new vat dye particularly recommended for producing blue-greys or full blacks on cellulosic materials, in particular on furnishing and curtain fabrics. Fastness figures on cotton include—Light 7, washing 5, chlorine 5.

INOCHROME COLOURS ON WOOL PIECE GOODS.—The Inochrome dyes are a range of premetallised dyes which are applied to wool in the usual way from a strongly acid dyebath. This card contains dyeings on wool piece of a range of twenty-five of these dyes, and the text includes details of the recommended dyeing methods and the fastness properties of the dyeings. The addition to the dyebath of Inochrome Salt P, Coptal BNB or BNA is advised to promote level dyeing, but no reduction in the amount of sulphuric acid employed is advocated even when using these products.

MODE SHADES ON WOOL–NYLON MIXTURES.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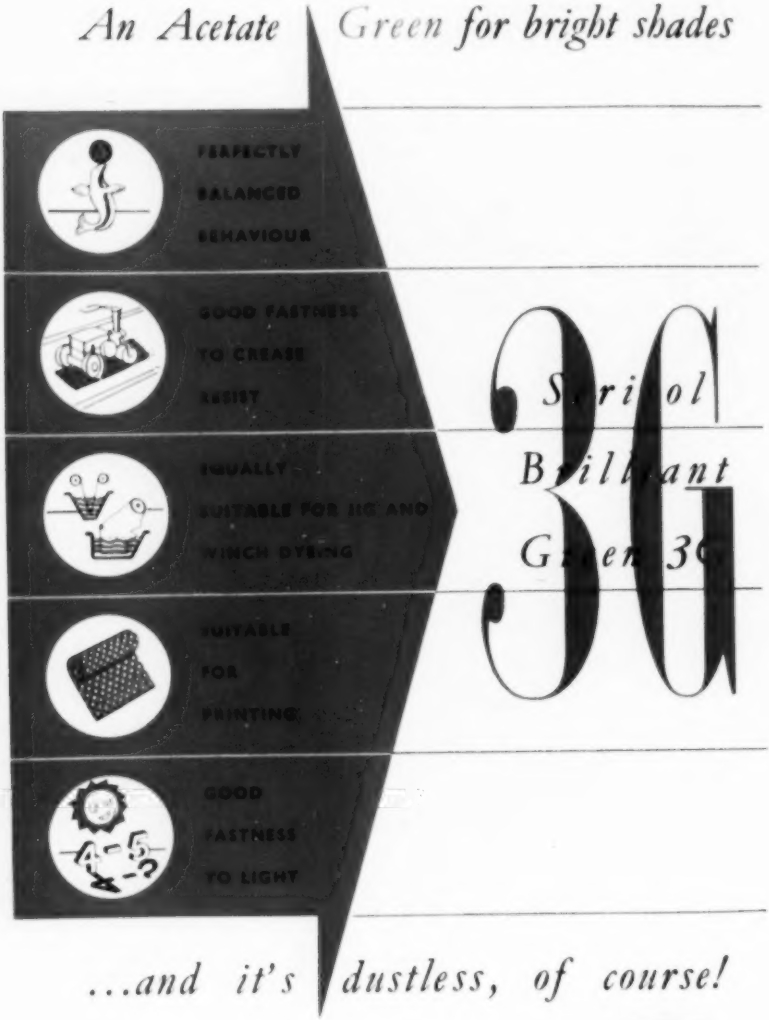
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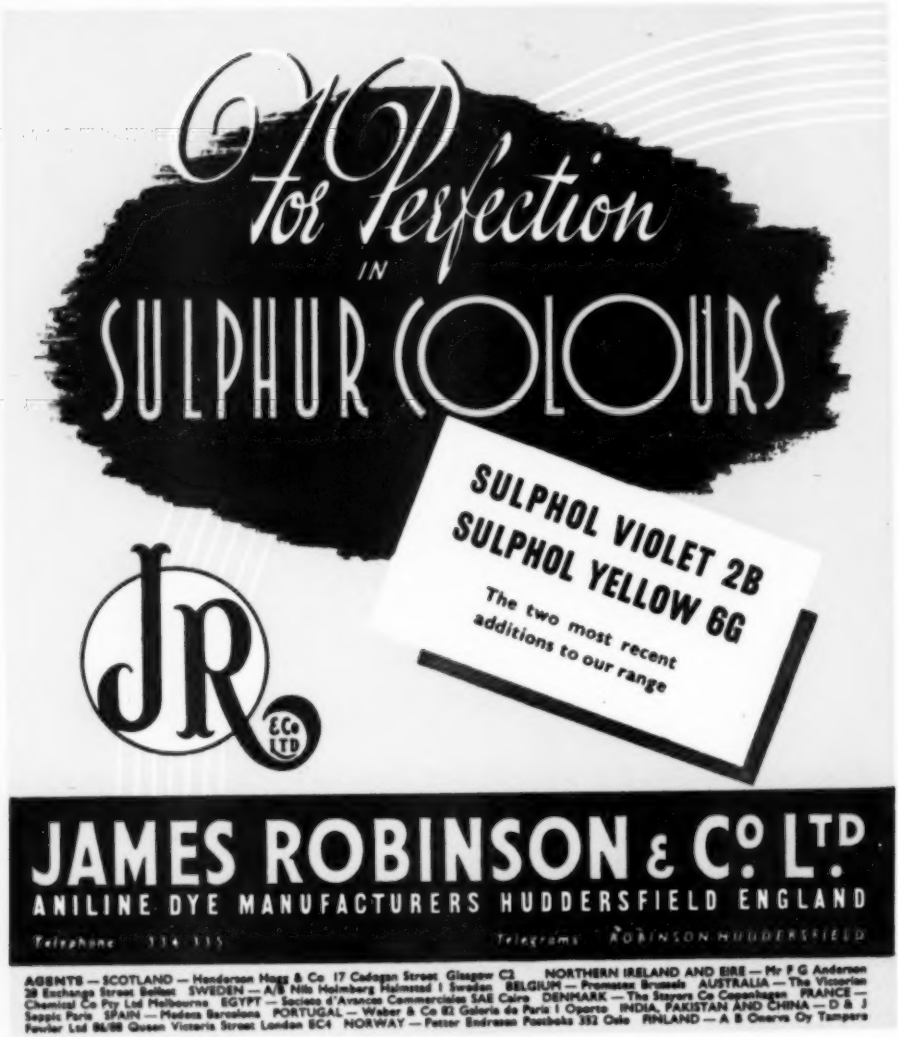
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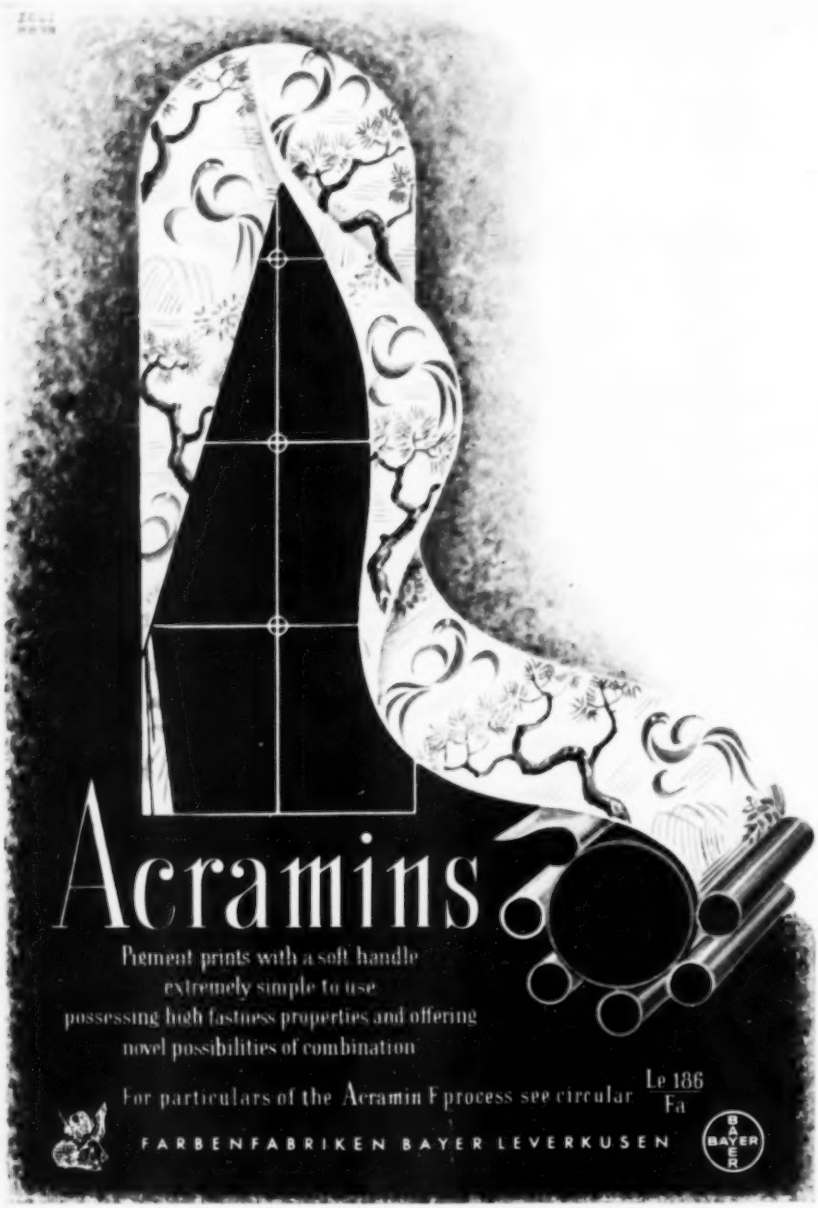
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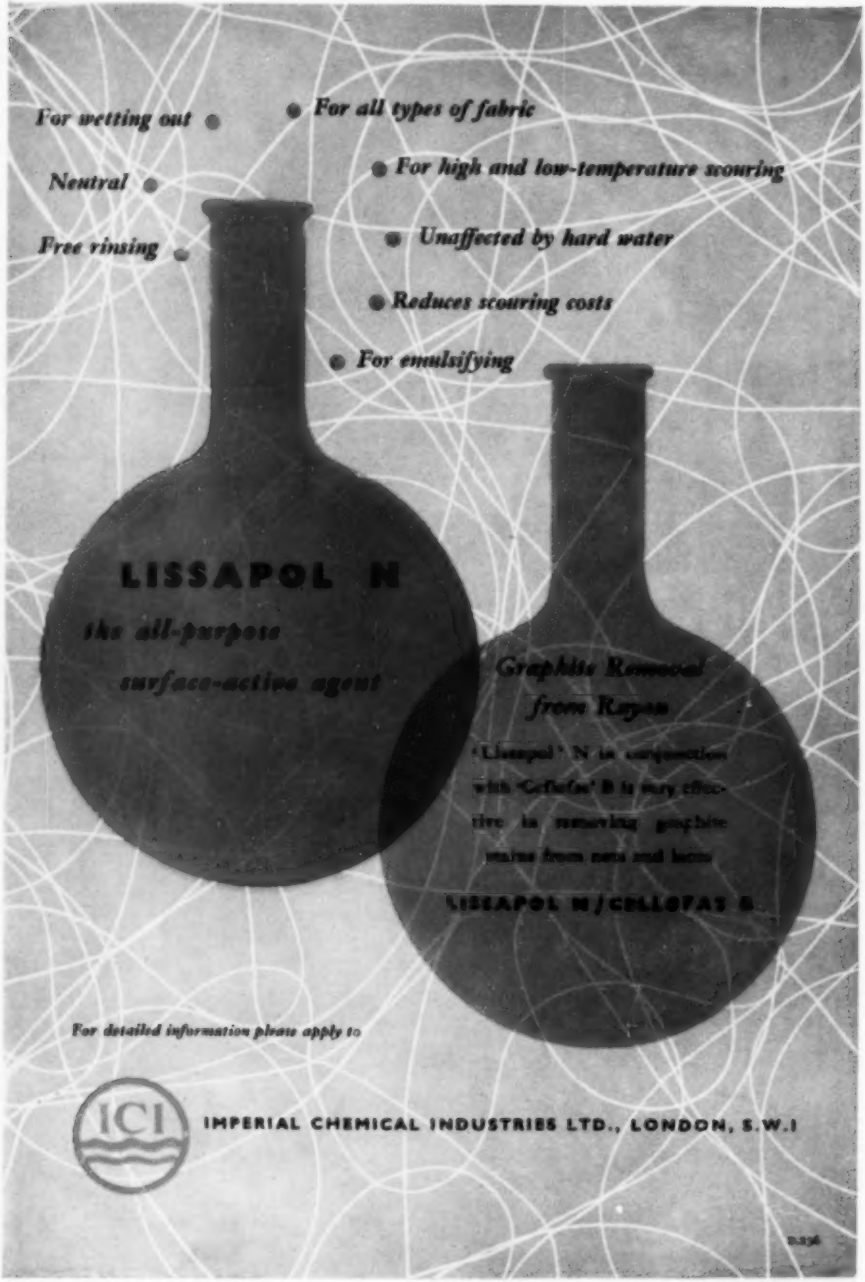
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DYEING ACETATE SILK AND MIXED FABRICS CONTAINING ACETATE SILK WITH INDIGOSOLS— This pattern card contains dyeings of a range of twenty-four solubilised vat dyes on acetate rayon piece material, sixteen mixture dyeings on the same material, eight mixture dyeings on acetate rayon–viscose rayon union, and a further eight mixture dyeings on acetate rayon–cotton union. The dyeing method employed is of particular interest, since it is not necessary to swell the acetate rayon prior to or during dyeing. A strongly acid dyebath (pH 1·4–2·5) containing Rongalite C is employed, and development is carried out in a solution of sulphuric acid and sodium nitrite in the normal way. A feature of this technique is that solid dyeings on unions of acetate rayon and cellulosic material can be readily obtained even in pale shades.

Farbenfabriken Bayer

INDANTHREN DYESTUFFS ON COTTON YARNS— This pattern card contains twenty-eight single and twenty-seven mixture dyeings on cotton yarn illustrating the range of Indanthren vat dyes. The text includes details of dyeing methods, the fastness properties of the individual dyes on cotton, and suggested uses for particular members of the series.

INDANTHREN BRILLIANT BLUE 4G POWDER— This vat dye gives bright greenish blues on all types of cellulosic material and may be applied by normal dyeing methods, pigment padding, pigment dyeing, and also the vat acid process. It is easily vatted and readily soluble, and is thus very suitable for dyeing in circulating-liquor machines. Dyeings are not fast to chlorine. Fastness grades on cotton include— Light 8, washing at 100°C. 4, chlorine 1.

INDANTHREN GREEN 4G POWDER— This vat dye is particularly recommended for producing yellowish-green dyeings having very good fastness to light, soda boiling, and hypochlorite bleaching. It is suitable for machine dyeing and pigment padding, and is also of interest in printing using the Rongalite–potash process. Fastness grades on cotton include— Light 7, soda boiling 4, chlorine 4–5.

INDANTHREN ORANGE GG PASTE— This vat dye is recommended for producing bright yellowish oranges of excellent fastness to light and to soda boiling–hypochlorite bleaching on cellulosic fibres. It is very level dyeing, and is thus of interest in combination shades. It may be printed by both the Rongalite–potash and the Colloresin processes, and is of interest for dyeing silk. It is also suitable for vulcanising treatments, a brand sufficiently free from copper and manganese being supplied for this purpose. Fastness figures include— Light 7–8, soda boiling 4–5, chlorine 5.

INDANTHREN PRINTING BROWN RL SUPRAFIX PASTE— This vat dye yields bright reddish browns on all types of cellulosic material when applied by either the Rongalite–potash or Colloresin process. It is of particular interest for use on curtain and furnishing fabrics. Fastness figures on cotton include— Light 6, washing at 100°C. 4–5, chlorine 5.

PHTHALOGEN BRILLIANT BLUE F3G— This is a phthalocyanine derivative developed for printing on all types of cellulosic fibres. The dye is prepared for printing by pasting it with Levasol P and then adding an aqueous solution of a copper or nickel salt. This mix is then added to the thickening, to which ammonium acetate has previously been added. The paste is printed, and the material then dried, steamed under neutral or acid conditions, and treated at 80–100°C. in a solution of formic acid or formic acid plus oxalic acid. Rinsing, soaping, and washing off complete the process. Using copper salts, bright blues are obtained, while nickel salts yield greenish blues. The stability of the printing pastes is limited, and decomposition is accelerated by heat and exposure to light. The new product may be direct-printed alongside vat dyes, Rapidogens, and most of the other types of dye used for printing. It is also of interest for printing silk. Fastness figures on cotton of prints produced using copper include —Light 7–8, washing at 100°C. 5, chlorine 5. Using nickel salts the corresponding figures are— Light 7–8, washing at 100°C. 5, chlorine 3–4.

Imperial Chemical Industries Ltd.

FIXANOL P— A FIXING AGENT FOR DIRECT COTTON DYESTUFFS— This Technical Circular describes the properties and uses of a new auxiliary product for after-treating dyeings of direct cotton dyes on cellulosic materials. Unlike the earlier product, Fixanol C, which did not improve the fastness of dyeings to alkaline wet treatments, Fixanol P does considerably increase the fastness to washing of a large number of direct dyes and also does not reduce the fastness to light or affect the hue of the dyeings to the same extent. The fastness of after-treated dyeings to acid and chrome cross-dyeing is, however, inferior to that of dyeings treated with the C brand. The text includes figures for the fastness to water, mild washing (both once and repeated, at 40°C.), alkaline perspiration, and light, of both untreated and after-treated samples; dyes which exhibit considerable improvement in particular respects are suitably marked.

FIXANOL P IN THE PAPER INDUSTRY— This pamphlet describes the use of this new auxiliary product in colouring paper, and suggests that advantages which result when it is present in the beater or applied as an after-treatment include increased dye retention, greater efficiency in sizing, particularly with deep dyeings, increased water fastness with most acid dyes, and an extension of the range of acid dyes which can be used for colouring both ordinary and absorbent papers.

BLEACHING AND DYEING OF ARDIL PROTEIN FIBRE AND ARDIL FIBRE UNIONS— This Technical Circular describes the dyeing behaviour and fastness properties of the whole range of acid and chrome dyes when applied to Ardil. The dyes have been classified into three groups— (1) dyes suitable for use on Ardil alone and in unions with other fibres; (2) dyes which may be used if absolutely necessary and provided dyeing is carried out below the boil; (3) dyes completely unsuitable for both Ardil and unions containing it. Particular attention is drawn to the capacity of this protein fibre for absorbing both acid and alkali and the consequent risk of unlevel, badly penetrated dyeings being produced when dyeing with acid milling and neutral-dyeing dyes or chrome dyes using the on-chrome or single-bath method. Special mention is made of the dyeing of Ardil–cellulosic fibre unions. Lists are given of direct dyes suitable for use under particular conditions and also of vat dyes which can be used for producing solid dyeings. If the cellulosic fibre is to be reserved or only slightly stained, selected chrome dyes or a mixture of three particular Ultralan dyes are recommended.

INTRODUCTION TO THE DYEING OF NYLON TEXTILES— As the subtitle indicates, this pamphlet is intended as a guide to students and others having a general interest in this subject. It includes an introduction and detailed general methods for applying the various classes of dye of major importance for dyeing nylon.

DYESTUFFS FOR JUTE— This pattern card contains dyeings on unbleached jute of fourteen basic, fourteen acid, and eleven direct cotton dyes. Details are given of recommended treatments prior to dyeing, the use of standing baths, and the methods of application of particular classes of dyes. Fastness figures for light and water are appended alongside each dyeing.

Yorkshire Dyeware & Chemical Co. Ltd.

SERISOL DYESTUFFS ON CELLULOSE ACETATE— This comprehensive pattern card contains dyeings on acetate rayon piece in three depths of shade of thirty-eight disperse dyes of the Serisol range; two diazotised and developed blacks and Hemasol AR Paste (a logwood derivative) are also illustrated. Some twenty-five pages of text include full details of the dyeing methods used and notes on the assessment of dyeing and fastness properties. The section dealing with the effect of burnt gas fumes is particularly detailed. A further valuable feature is that in the case of each dye information is given as to whether it is homogeneous or a mixture and the chemical class to which it belongs. A very full selection of fastness data and a summary of the dyeing properties of each dye are appended alongside the relevant patterns.

Abstracts from British and Foreign Journals and Patents

(The titles of abstracts may be modified. Abbreviations of names of firms are listed in J.S.D.C., **68**, *23 (Jan. 1952); while other abbreviations and symbols, together with a list of the periodicals abstracted, will be found at the end of the annual index.)*

I—PLANT; MACHINERY; BUILDINGS

Action of Dilute Inorganic Acids on Stainless Steel. C. F. Poe and R. M. Malcom. *Ind. Eng. Chem.*, **43**, 2572–2575 (Nov. 1951).

Experiments are described to assess the effect of some common inorganic acids on 18–9 chromium–nickel stainless steel. Sheets of the steel are exposed to the action of 1, 0·1, and 0·01 N. acids for ten weeks at 25°C. In duplicate experiments the sheets in contact with acid are boiled for 30 min. daily. The change in weight of each sheet is determined frequently, and the amounts of Fe, Cr, and Ni dissolved by the acids are estimated at the end of two and ten weeks by Snell's procedure. The only acid active on the stainless steel at 25°C. is N-HCl. Boiling greatly increases the action of N-HCl, N-HBr, N-H_2SO_4, and N-H_2SO_3. Very little action is found with boric, nitric, phosphoric, phosphorous, and selenious acids; it is therefore considered that stainless steel utensils may be used when these acids are present in dilute solutions. There are three tables showing experimental data and a list of references. A. H.

PATENTS

Wrappers for Rayon Cakes. American Viscose Corpn. *BP* 663,931

A wrapper for protecting rayon cakes during wet processing consists of a thin fibrous sheet comprising 7–70% of saponifiable cellulose ester fibres in combination with fibres not affected by aqueous saponifying agents, the fibres being boiled together in a condition of unbalance such that, when the wrapped cake is washed or dried after complete or part saponification of the ester fibres in the wrapper, the latter shrinks on the cake. This enables the wrapped cake to be handled without disturbing the surface of the cake. C. O. C.

Coating Wire or the like. Redding Manufacturing Co. *BP* 663,518

Treating Continuous Lengths of Textiles with Liquids. American Viscose Corpn. *BP* 662,980

Yarn or fabric in rope form is repeatedly immersed in the processing liquor as the yarn or fabric passes along a helical path about a rotating surface, e.g. a roll, which is partly submerged in the bath. Between immersions the yarn or fabric passes about a rotating member having spaced axially extending ridges, a large part of the liquor in the material being then removed from it before it is again immersed. C. O. C.

Rigging and Plaiting Fabrics. Sellers & Co. (Huddersfield). *BP* 663,636

Back Greys. Eddystone Manufacturing Co. *BP* 664,313

A woven nylon back grey is described. It has the advantages that it is dimensionally stable during washing and drying, so that its absorbency remains constant throughout its working life. C. O. C.

Screen Printing Table. P. F. van Vlissingen & Co.'s Katoenfabrieken. *BP* 662,181

The table top is made of heavy material, e.g. concrete, in which is embedded at least one heating element, preferably of serpentine or zig-zag form. This gives good heating of the table top with little or no deformation thereof. C. O. C.

Pneumatic Devices for Applying Short-staple Flock to Fabrics. Heberlein & Co. *BP* 662,481

A machine is described in which, by controlling the vibration frequency and amplitude of vibration of the lacquered fabric, considerably improved flocking is obtained. The lighter the individual fibres in the flock, the lower the vibration frequencies and amplitudes required. C. O. C.

Gearing and Brake Mechanism for Wet and Dry Cleaning Machines. Sutcliffe, Speakman & Co. *BP* 663,941

Control Apparatus for Driers. Robertshaw–Fulton Controls Co. *BP* 662,437

A control especially suitable for power-driven clothes driers is described. C. O. C.

Garment Pressing. R. G. Whitaker. *BP* 664,307

A steam-heated hot-plate serves as the outer wall of a steam chest through which passes a steam discharge tube from the main supply. Steam passed through it is discharged through jets pointing back at the hot-plate surface, thus ensuring that it is dry. Around the whole is a perforated metal shell, over which is placed the garment to be dry-steamed. J. W. B.

Ironing Machines. Wall Engineering Co. *BP* 662,461

Dyeing Hides and Skins. Concerie Alta Italia di Giraudo, Ammendola & Pepino. *BP* 664,059

The skins are placed on an endless wire mesh belt. A projector–photoelectric cell unit and several sprayers spaced apart are moved synchronously with one another perpendicularly to the direction of movement of the belt. The cell receives light from the projector only when the cell comes in front of a part of the web not covered by a skin. The impressions received by the cell are recorded on a repeating device which moves synchronously with the endless conveyor, the record being used to operate the various sprayer pistols so that they function only when a skin comes in front of them. C. O. C.

II—WATER AND EFFLUENTS

PATENT

Regeneration of Base-exchange Material. Czechoslovak Metal & Engineering Works. *BP* 660,380

The regenerating liquor is fed in before the whole column of base-exchange material is exhausted, and that part of the material which has been completely exhausted is regenerated separately from that which has been only partly exhausted. This enables a saving of up to 30% of the regenerative material previously required. C. O. C.

III—CHEMICALS; AUXILIARY PRODUCTS; FINISHING MATERIALS

Action of Antifoaming Agents at Optimum Concentrations. S. Ross and G. J. Young. *Ind. Eng. Chem.*, **43**, 2520–2525 (Nov. 1951).

An attempt has been made to find the mechanisms underlying the actions of foam-inhibiting agents. To compare the effect of different agents the optimum concentration of each has been determined, and the antifoaming effect compared with the viscosity, the spreading coefficients, and the entering coefficients of each agent for each of two aqueous foaming solutions. Two distinct effects of foam inhibitors could be distinguished— In one case the agent promotes liquid drainage from the foam films without much affecting the final thickness of the film at which its rupture takes place; this effect is believed to be the result of a decreased surface viscosity produced by presence of the agent. In the other case the agent causes rupture of thick liquid films before drainage has thinned them to breaking point. Many agents produce combinations of the two effects. Details are given of methods of measuring initial foam density, foam stability, surface and interfacial tensions, and viscosity. The two foaming systems used in the experiments are (*a*) 0·50% by weight Nacconol NRSF and 0·75% by weight sodium silicate solution, technical grade; (*b*) 0·20% by weight Aerosol OT 100% pure and 1·00% by weight glycerol. The sixteen antifoaming agents tested include tributyl phosphate, nonyl Cellosolve, and silicone oil. There is a discussion of the mechanism of antifoaming action, six tables and three graphs showing rate of escape of liquid from foams, rate of escape of gas from foams, and variation of density of foam with time. A. H.

Structure and Rôle of Wetting Agents— IV. D. G. Dervichian and F. Larchampt. *Bull. Soc. chim. France*, 289–294 (1951); *Chem. Abs.*, **45**, 9980 (10 Nov. 1951).

The usual explanations of wetting, based exclusively on the conceptions of surface tension and contact angle, do not account for the essential details of the phenomenon. Wettability depends not only on the nature of the liquid but also on that of the surface being wetted. For a liquid to adhere to a surface, the free energy of adhesion must exceed that of cohesion. For a liquid to spread spontaneously on a surface, not only must the free energy of adhesion be great, but also the molecules of the surface must be unorganised and mobile. The action of various wetting agents is explained. C. O. C.

Nitroalkane Derivatives related to DDT. H. B. Hass, M. B. Neher, and R. T. Blickenstaff. *Ind. Eng. Chem.*, **43**, 2875–2878 (Dec. 1951).

The synthesis and properties of a number of diarylnitroalkanes and their chlorinated derivatives are described. Many of these compounds are more effective insecticides than DDT, in particular 1:1-bis-*p*-chlorophenyl-2-nitropropane and the corresponding nitrobutane are about five times as effective as DDT and only half as toxic to warm-blooded animals. W. K. R.

PATENTS

Germicidal Detergent. General Aniline. *BP* 663,565

A mixture of an alkaline inorganic detergent (1–4 parts by weight), a non-ionic polyglycol ether detergent (1), and a quaternary type bactericide (not $< 0{\cdot}1\%$ by weight of the other two components) is used as a combined detergent and germicidal composition. C. O. C.

Carding Lubricant. Texaco Development Corpn. *BP* 663,576

The fibres are treated before carding with 0·2–0·4% by weight of refined light mineral lubricating oil of SUS viscosity 36–75 sec. at 100°F. and 5–20% on the weight of the oil of rosin or a partly hydrogenated rosin. This gives good lubrication without softening the fibres. C. O. C.

Antistatic Compositions. Courtaulds. *BP* 662,542

Hydroxyalkylamine salts of dibasic acids of formula $HOOC{\cdot}(CH_2)_n{\cdot}COOH$ ($n > 3$), *e.g.* triethanolamine sebacate, are good antistatic agents for use on textiles. C. O. C.

Concentrated Emulsions remaining Stable on Dilution with Water. H. Woudhuysen. *BP* 663,725

Several incompatible components, either alone or dissolved or dispersed in non-aqueous solvents, are successively mixed with one another, the disturbance of miscibility caused by each addition being counteracted by adding soap and/or soap-forming acid and/or alkali, the amounts used being such as are required to prevent breakdown of the emulsion on addition of water. C. O. C.

Hydrophilic Cellulose Gel. Anglo International Industries. *BP* 663,621

Fibrous cellulose material is treated with 18–40% aq. NaOH or KOH using at least 5 litres of lye per kilogram of dry cellulose at not $> 20°$C. Then 35–40% of water on the weight of the whole is removed, and the whole heated to 40–70°C. under 5–10 atm. until a depolymerised cellulosic gel, from which the fibrous structure cannot be regenerated, is obtained. The product can replace cellulose esters and ethers for many purposes.

BP 663,622

Similar products are obtained by heating cellulose containing 50–60% water at 70°C. under 5–8 atm. with 20–30% (on the weight of the final product) of an alkali-metal hydroxide or carbonate, preferably in presence of iron oxide and/or alumina. C. O. C.

Cationic Urea–Formaldehyde Resins having Affinity for Cellulose. British Industrial Plastics. *BP* 663,736

The product obtained by treating a condensate of 2–3 mol. of H·CHO and 1 mol. of urea with 2–80% by weight of a polyfunctional amine at pH < 4 is water-soluble, and when dry is thermosetting. It is substantive to cellulose, and may be used for imparting wet strength to paper, for imparting dimensional stability to cellulosic textiles, and for finishing leather. C. O. C.

Condensates from Aminotriazines and *N*-Hydroxymethylamides. Ciba. *BP* 662,548

A hydroxymethyl compound of an aminotriazine or of a derivative thereof, or an ether of such a compound with an aliphatic monohydric sat. alcohol containing > 5 C, is treated with a hydroxymethylamide, containing the group $-CO{\cdot}N(CH_2OH)-$, in which at least one of the two residues to which this group is bound contains at least 1 C. The reaction is carried out in an organic solvent in such a manner that the readily volatile hydroxy compounds formed are continuously removed from the reaction mixture. The products have wide uses as assistants in the textile, paper, and leather industries, *e.g.* as water-repellent, crease-resistant, or shrink-resistant agents. C. O. C.

Styrene Copolymers— Sizes, Paper Coatings, etc. Monsanto. *BP* 663,841

Copolymers of compounds of formula $R{\cdot}CH{:}CH_2$ (R = subst. or unsubst. phenyl radical) with monofunctional secondary alkyl half esters of ethylenedicarboxylic acids are used as sizes, for coating paper, etc. C. O. C.

Inorganic Oxide Aquasols. Monsanto. *BP* 663,013

Concentrated inorganic aquasols are prepared by passing a mixture of an alkaline-reacting inorganic oxide aquasol containing alkali-metal ions and up to 45% by wt. of the same oxide in ionic form or as particles of < 40 mμ. mean size, *e.g.* dil. sodium silicate soln., through a cation-exchange bed until the effluent is slightly below neutral pH. The effluent is made alkaline with a compound capable of yielding alkali-metal, ammonium, or quaternary ammonium ions, *e.g.* by adding more sodium silicate soln., heating to above 100°C., and recycling through the exchange with alkali-metal ions and up to 4% by wt. of the same inorganic oxide. Silica aquasols thus prepared have relatively large particle size and are useful as spinning auxiliaries. J. W. B.

Starch Compositions. S. C. Johnson & Son. *BP* 662,944

A fabric-treating composition comprises wax, a water-soluble multivalent metal salt, a protective colloid, raw starch to 50–98% by wt., and water. In one example water (62·2 parts by wt.) is heated to approx. 185°F., and hide glue (5·2) is dispersed in it. Commercial 22° Al formate solution (16·3) and paraffin wax (m.p. 130–132°F.) (16·3) are added and dispersed. To this is added a paste from raw maize starch (100) containing no oils and water (100), followed by cold water (700), and the mixture is made homogeneous. Before treating washed laundry fabrics, a further dilution with 5 vol. of cold water is effected. Crisp, starchy, stain-resistant, and water-repellent finishes are given.

BP 662,945

A fabric-treating composition comprises wax, *e.g.* paraffin wax, a water-soluble multivalent metal salt, *e.g.* aluminium formate, cooked and raw starch totalling 50–98% by wt. and containing at least 3 parts raw to 1 cooked, and water. Protective colloids, *e.g.* polyvinyl alcohol, may be added. J. W. B.

Chemically Modified Starches— Sizing and Finishing Agents. United States Rubber Co. *BP* 663,358

Starch, either of the cold water-insoluble or water-soluble variety, reacts with vinyl sulphone $(CH_2{:}CH)_2SO_2$ to form products which are insoluble in boiling water. 0·1–50% Vinyl sulphone on the weight of the starch may be used. This reaction may be utilised to reduce the viscosity of starch pastes, to set starch gels, to modify starch adhesives, or to produce permanent sizing or finishes in fabrics by treating them with starch and then with vinyl sulphone. C. O. C.

Methylpolysiloxanes. Rhône-Poulenc. *BP* 661,737

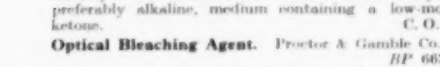
Liquid methylpolysiloxanes which can be stored for long periods without change in viscosity, and which are useful for waterproofing textiles, etc., are obtained by hydrolysing in ethereal solution methylhalogenosilanes of formula $(CH_3)_nSiX_{4-n}$ having a $CH_3{:}Si$ ratio $< 1{\cdot}4$, distilling the resulting resin under reduced pressure, and separating the fractions which (*a*) have a $CH_3{:}Si$ ratio $< 1{\cdot}4$, (*b*) have 8–20 Si atoms in the polysiloxane molecule, (*c*) boil at 120–250°C. under 8 mm. pressure, and (*d*) gel under the action of lead naphthenate. W. G. C.

Acyl Derivatives of Aminostilbenesulphonic Acids—Optical Bleaching Agents. American Cyanamid Co. *BP* 662,558

Good yields of a pure product having remarkable optical bleaching effects on cellulosic materials are obtained by treating 4:4′-diaminostilbene-2:2′-disulphonic acid with a carboxylic acid halide of the benzene series in aqueous, preferably alkaline, medium containing a low-mol.wt. ketone. C. O. C.

Optical Bleaching Agent. Procter & Gamble Co. *BP* 663,919

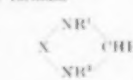
4 : 4′-Di-*p*-methoxybenzoylaminostilbene-2 : 2′-disulphonic acid and its salts, e.g.—



are highly efficient optical bleaching agents having affinity for cellulose and resistant to sodium hypochlorite. C. O. C.

Gas-fading Inhibitor. ICI. *BP* 664,204

Compounds of formula—



(R^1 and R^3 = Ar or aralkyl; R^2 = H, Alk, or Ar; X = alkylene of > 1 C), e.g. 1:3-diphenylimnazolidine, are gas-fading inhibitors which are fast to light, washing, and dry cleaning. They can be applied in the dyebath. C. O. C.

***NN′N′*-Triphenylguanidine as Gas-fading Inhibitor.** ICI. *BP* 664,079

NN′N′-Triphenylguanidine is a gas-fading inhibitor which may be used in the dyebath. C. O. C.

IV—RAW MATERIALS; INTERMEDIATES; COLOURING MATTERS

Chemical Engineering Unit Processes Review. *Ind. Eng. Chem.*, **43**, 1803–2052 (Sept. 1951).

Topics reviewed comprise kinetics and equilibria, alkylation, amination by reduction, ammonolysis, combustion, esterification, fermentation, Friedel–Crafts reactions, halogenation, hydrogenation and hydrogenolysis, nitration, oxidation, polymerisation, pyrolysis of coal and shale, pyrolytic and catalytic decomposition of hydrocarbons, and sulphonation and sulphation. C. J. W. H.

Catalytic Air Oxidation of Higher Aromatics to Phthalic Anhydride. C. R. Kinney and I. Pincus. *Ind. Eng. Chem.*, **43**, 2880–2884 (Dec. 1951).

Phthalic anhydride of satisfactory quality was obtained in 75% yield by vapour-phase air oxidation of a crude coal-tar 200–235°C. fraction using a silica-based vanadium pentoxide catalyst. Pure methylnaphthalene and a methylnaphthalene coal-tar fraction (235–270°C.) gave 28–40% yields, free from methyl derivatives. Using an alumina-based catalyst, anthracene, phenanthrene, and commercial "anthracene salts" gave yields of 50, 35, and 24% respectively. W. K. R.

Relation between Constitution and Fine Structure in the Absorption Spectra of Aromatic Hydrocarbons. G. Kortüm and G. Dressen. *Chem. Ber.*, **84**, 192–203 (1951): *Chem. Abs.*, **45**, 9491 (10 Nov. 1951).

Previous work is reviewed to bring out the pertinent conclusions—(1) Increasing the number of substitutions in C_6H_6, increasing the chain-length of an alkyl substituent, or presence of several independent chromophores decreases the fine structure ("negative St-effect"). (2) Cyclisation of *ortho* substituents (as in indane) gives a positive St-effect. (3) Conjugation of previously isolated chromophores produces an entirely new chromophore with a new absorption spectrum not comparable with the preceding. Conjugated compounds in general show no fine structure (F.S.), but the polyenes are exceptional in showing F.S. with increasing length of the conjugated chain. (4) Steric hindrance of coplanarity in $(C_6H_5)_2$ derivatives causes a negative St-effect, closely related to (1) above. Two tables and 14 absorption curves show the data for toluene (I), $(C_6H_5)CH_2$ (II), $(C_6H_5)_2CH$ (III), $(C_6H_5)_3C$ (IV), $(C_6H_5)_2CH_2$ (V), diphenyl (VI), triphenylamine (VII), $(>C(C_6H_5)_2)_2$ (VIII), $(CH_2{:}C(C_6H_5)_2)_2$ (IX), $(CH{\cdot}CH{\cdot}CH{\cdot}C(C_6H_5)_2)_2$ (X), $(\cdot CH{\cdot}CH{\cdot}CH{\cdot}CH{\cdot}C(C_6H_5)_2)_2$ (XI), $(CH{\cdot}CH{\cdot}CH{\cdot}CH{\cdot}CH{\cdot}C(C_6H_5)_2)_2$ (XII) all dissolved in *cyclo*hexane and II and VI as vapours. A small but systematic negative St-effect is shown by I, II, III, and V as the C_6H_5 groups increase. IV is exceptional with no F.S. because the high-frequency band is strongly displaced toward the red, probably as a result of the steric interaction of the four phenyl groups. Theories previously proposed to explain the relations between F.S. effects and constitution—(1) inter- and intra-molecular Stark effect, (2) planarity effect, (3) "loose-bolt" effect (cf. Lewis and Calvin, *Chem. Abs.*, **34**, 671 (1940))—are discussed and discarded. A theory is advanced in which molecules whose parts can twist with respect to each other around an axis develop torsional vibrations, which must attain a certain maximum energy before any "free" rotation around the axis occurs. A rigid molecular model must show F.S. Whenever torsional vibrations allow the excited states of different chromophores to exist together, the lifetimes of these excited states are shortened and the F.S. is lost; e.g. C_6H_5, $C_{10}H_8$, etc. are rigid and show F.S., but diphenyl undergoes torsion sufficiently to disturb its conjugation before any actual free rotation occurs. Thus the excited state of its molecule has the excited C_6H_5 chromophore overlapping the excited $(C_6H_5)_2$ chromophore, the lifetime of the excited state is short, and no F.S. is shown. The rigid molecules mentioned are planar, but the rigidity and not the planarity is responsible for F.S. Diphenyl is also planar but not rigid, and its spectrum has no F.S. The disappearance of F.S. in the absorption of tetraphenylpolyenes (VIII–XII) as compared with diphenylpolyenes is attributed to the action of two of the four C_6H_5 groups as mere "ballast" to increase the moment of inertia of the molecule, and give greater ease of excitation for the twist of the molecule around the single bonds, thus causing the excited state to consist of various excited chromophores (all or various parts of the conjugated chain) as in the case of diphenyl. Many examples from the literature are discussed on the basis of this theory. C. O. C.

Hygroscopic Properties of U.S.A. Certified Dyes. C. Stein. *J. Assocn. Off. Agric. Chem.*, **34**, 432–436 (1951): *Chem. Abs.*, **45**, 9865 (10 Nov. 1951).

The hygroscopic properties of 18 certifiable (U.S.A.) dyes are given. The water-soluble dyes readily absorb water, the water-insoluble ones do not. Water-absorption was approximately the same at 25° and 35°C. The method used was to dry the dye at 135°C., place in a closed chamber of controlled humidity (20, 40, 60, 80, and 100% R.H.), and allow the weight to become constant (in about three weeks). C. O. C.

Synthesis of Certain Trifluoromethyldiphenyl Derivatives. M. R. Pettit and J. C. Tatlow. *J.C.S.*, 3459–3464 (Dec. 1951).

A new preparation of the known 3-trifluoromethyldiphenyl (cf. Bradsher and Bond, *J. Amer. Chem. Soc.*, **71**, 2659 (1949)) is described, together with a series of its derivatives containing trifluoromethyl groups, viz. the nitro and amino compounds, and some symmetrical dinitro- and diamino-bistrifluoromethyldiphenyls. H. H. H.

Some Furoyl Azo Dyes VI—Disazo Dyes from Methylfuroylacetarylides. R. Andrisano and D. dal M. Casoni. *Boll. Sci. Facoltà Chim. ind. Bologna*, **8**, 77–78 (1950): *Chem. Abs.*, **45**, 9865 (10 Nov. 1951).

At 5°C. a soln. of 0·05 mol. of tetrazotised benzidine couples with an alkaline (0·4 mol. NaOH) soln. of 0·1 mol. of R·CH_2·CO·NH·Ar (R = methylfuroyl) to yield dyes of formula [R·CH(CO·NH·Ar)·N_2·C_6H_4]$_2$. C. O. C.

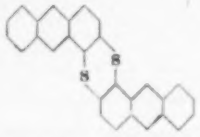
Reactions of Methazonic Acid. II—Some 3-Aminolepidines, 3-Amino-4-arylquinolines, and Derived Compounds. K. Schofield and R. S. Theobald. *J.C.S.*, 2992–2995 (Nov. 1951).

As a group, 3-aminoquinolines have hitherto most frequently been prepared by methods other than the reduction of 3-nitroquinolines, and now several 3-aminolepidines and 3-amino-4-arylquinolines are reported by reduction of the related 3-nitroquinolines (cf. Part I, *ibid.*, 395 (1950)). 3-Aminolepidine on diazotisation and

treatment with pyridine gives two isomeric 3-pyridyllepidines. 3-Amino-4-phenyl- and 3-amino-4-*p*-methoxyphenyl-quinoline deaminate to 4-phenyl- and 4-*p*-methoxyphenyl-quinoline. 3-Benzamido-4-phenyl-quinoline is converted by P_2O_5 into 2-phenyl-3:4-5:6-dibenzo-1:7-naphthyridine, the quaternisation of which is reported. H. H. H.

Anthracene Khaki. H. Hiyama, A. Ito, and M. Noguchi. *J. Chem. Soc. Japan, Ind. Chem. Sect.*, **52**, 198–201 and 252–256 (1949): *Chem. Abs.*, **45**, 7091 (25 Aug. 1951).

The fusion of anthracene and sulphur to give Anthracene Khaki was studied to determine the effect of temperature (250–360°C) and time (4–6 hr.) on the hue and the amount of sulphur in the product. After purifying and drying at 100°C. analysis gave $C_{14}H_{14}O_2S_3$, losing $5H_2O$ at the b.p. of xylene. Various chlorine derivatives of anthracene were synthesised and fused with sulphur in an attempt to find where the S atoms link with the anthracene mol. For the same reason several simple sulphur derivatives of anthracene, e.g. 1:4:5:8-tetramercaptoanthraquinone, were examined for Na_2S-vat-forming property and affinity for cellulose. From these results and sulphur fusion of the dinaphthothianthren—



it was deduced that this compound is the parent body of Anthracene Khaki. R. K. F.

Second Century B.C. Rose Madder from Corinth and Athens. *Amer. J. Archaeol.*, **55**, 236–239 (1951): *Chem. Abs.*, **45**, 9889 (10 Nov. 1951).

Microscopic examination of ancient pigment indicated madder. Al was confirmed spectroscopically and by precipitation as $Al(OH)_3$. The reflectance curve was similar to that of modern rose madder. C. O. C.

Polynuclear Heterocyclic Systems. III — The 3:4-Benzacridine–5:10-Dihydro-3:4-benzacridine Complex. G. M. Badger, J. H. Seidler, and B. Thomson. *J.C.S.*, 3207–3211 (Nov. 1951).

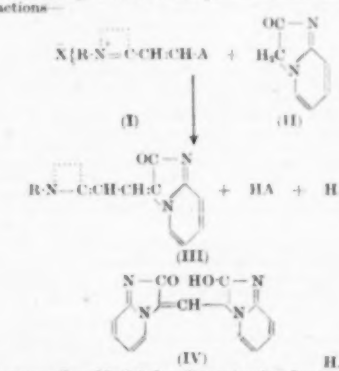
Lithium aluminium hydride reduces 3:4-benzacridone to give an orange molecular complex of 3:4-benzacridine and its 5:10-dihydro derivative; it also converts phenanthridone and 9-chlorophenanthridine to 9:10-dihydrophenanthridine, which dehydrogenates over palladium to phenanthridine in good yield. Similarly, 3:4-benzocinnoline is obtained by reduction of its *N*-oxide, and also, in one step, by reduction of 2:2′dinitrodiphenyl, both with lithium aluminium hydride. H. H. H.

Colour of Organic Compounds. V— Thermochromic Spirans. E. B. Knott. *J.C.S.*, 3038–3047 (Nov. 1951).

The factors affecting the thermochromic behaviour of certain colourless spirans containing one or more pyran residues are analysed. It is shown that, whereas the case of intramolecular ionisation resulting in dye formation increases with an increasing electron-donating ($-M$) effect of the heterocyclic *spiro* component (cf. Wizinger and Wenning, *Helv. Chim. Acta*, **23**, 247 (1940)), it may be affected in either direction by changes in the $+M$ effect of the ketonic fragment resulting from the ring-opening, and is largely independent of the degree of degeneracy of the resonance system responsible for the colour of the dye. The inhibitory effect of the introduction of substituents into certain positions of the pyran rings is explained on the basis of steric hindrance to planarity of the dye molecules. Certain colour phenomena associated with Wizinger's dyes are explained on the basis of their strongly dipolar nature. H. H. H.

Abnormalities of Dyes derived from 3:7*a*-Diazaindan-2-one. E. B. Knott. *J.C.S.*, 3033–3038 (Nov. 1951).

Dimethin*meso*cyanines are obtained by condensation of a 2-substituted vinyl derivative of a cyclic quaternary ammonium salt (I) with a ketomethylene compound in the presence of a base; the group A is usually an anilino, acetanilido, or alkylthio residue. 3:7*a*-Diazaindan-2-one (II) is here employed as the ketomethylene compound, and the dimethin*meso*cyanines (III) produced show a hypsochromic absorption shift on increasing the solvent polarity, while being reactive towards cyclic ketomethylene compounds and so forming oxonols. When the diazaindanone is used in excess, the formation of bis-(3:7*a*-diaza-1-indan-2-one)methinoxonol (IV) is observed. The reason for the strongly dipolar nature of the dyes (III) is discussed, together with the possible mechanism of the reactions—



(I) (II) (III) (IV)

H. H. H.

Electron Gas Model for Quantitative Interpretation of Light Absorption by Organic Dyes— II. Part B —Disturbance of the Electron Gas by Hetero Atoms. H. Kuhn. *Helv. Chim. Acta*, **34**, 2371–2402 (Dec. 1951).

The model assumes that π electrons can move freely along a polymethin chain. Replacement of CH by electronegative N leads to stabilisation of π electrons, with consequent depression of the energy level. The relative distance between the normal energy state and the higher energy level is thus altered, and a change in the absorption maximum occurs. Replacement of CH by N in symmetrical polymethins results in displacement of the absorption maximum (i) to longer wavelengths if the number of double bonds (j) along the mesomeric chain is odd, (ii) to shorter wavelengths when (j) is even. Mathematical treatment of experimental results leads to a quantitative interpretation of absorption differences when comparisons are made between cyanines and azacyanines, diphenylmethane dyes and corresponding indamines, xanthens and oxazines, and acridine dyes and azines.

G. L.

Acetylenic Dyes— Comparison with Corresponding Ethylenic Dyes. C. Dufraisse, A. Etienne, and P. Barbieri. *Compt. rend.*, **232**, 1977–1980 (1951): *Chem. Abs.*, **45**, 9367 (10 Nov. 1951).

Derivatives of $(C_6H_5)_2C(OH)\cdot C_6H_5$, $(C_6H_5)_2C(OH)\cdot CH{:}CH\cdot C_6H_5$, and $(C_6H_5)_2C(OH)\cdot C{:}C\cdot C_6H_5$ were compared. The following primary and secondary absorption bands (in Å.) were obtained, where R = *p*-$(CH_3)_2N\cdot C_6H_4$—: R·C(OH)$(C_6H_5)_2$ 4650, 3450; R·C(OH)(C_6H_5)·CH:CH·C_6H_5 5050, 3400; $(C_6H_5)_2C(OH)\cdot CH{:}CH\cdot$R 5150, 3400; R·C(OH)·$(C_6H_5)$·C:C·$C_6H_5$ 4900, 2710; $(C_6H_5)_2C(OH)\cdot C{:}C\cdot$R 5100, —; $R_2C(OH)\cdot C_6H_5$ 6200, 4280; $R_2C(OH)\cdot CH{:}CH\cdot C_6H_5$ 6560, 4900; R·C(OH)(C_6H_5)·CH:CH·R 7150, 4625; $R_2C(OH)\cdot C{:}C\cdot C_6H_5$ 6900, 4890; R·C(OH)(C_6H_5)·C:C·R 7360, 4775; $R_2C\cdot OH$ 5870, (3550, 3750); $R_2C(OH)\cdot CH{:}CH\cdot$R 6900, (4000, 4200); $R_2C(OH)\cdot C{:}C\cdot$R 6700, (3950, 4150). The acetylenic group is thus sometimes more and sometimes less powerful than the ethylenic group in shifting absorption bands. C. O. C.

Purpurogallin. IX — Structure of the Isomeric *O*-Methyl Ethers of *β*-Methyltropolone. P. Akroyd, R. D. Haworth, and J. D. Hobson. *J.C.S.*, 3427–3429 (Dec. 1951).

In Part IV (*ibid.*, 561 (Feb. 1951)) two *β*-methyltropolone methyl ethers, m.p. 97° and 46°C., were described and referred to as the methyl ethers -A and -B respectively.

The methoxyl group has now been found readily replaceable by the amino, anilino, or hydrazino group. The Stevens-McEadyen decomposition reaction has been applied to the hydrazino derivatives -A and -B, and the products have been reduced to 3- and 4-methylcycloheptanone respectively; in consequence the -A and -B series are assigned structures I and II—

(I)

(II)

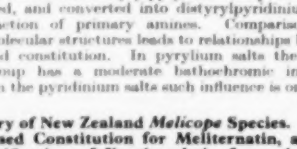
H. H. H.

Distyrylpyrylium and Distyrylpyridinium Salts. R. Winzinger and K. Wagner. *Helv. Chim. Acta*, **34**, 2290–2301 (Dec. 1951).

A number of 2:6-distyrylpyrylium compounds were synthesised, and converted into distyrylpyridinium salts by the action of primary amines. Comparison with similar molecular structures leads to relationships between colour and constitution. In pyrylium salts the second styryl group has a moderate bathochromic influence, whereas in the pyridinium salts such influence is only very weak. G. L.

Chemistry of New Zealand *Melicope* Species. VI—A Revised Constitution for Meliternatin, and the Identification of Xanthoxyletin from the Bark of *Melicope ternata*. L. H. Briggs and R. H. Locker. *J.C.S.*, 3131–3136 (Nov. 1951).

Evidence is now given which supports the formulation of meliternatin as 3:5-dimethoxy-6:7-3':4'-bismethylenedioxyflavone—



"Compound E", the non-flavone substance isolated from *Melicope ternata*, has been identified as xanthoxyletin—

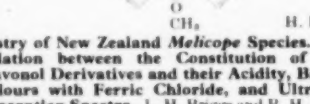
H. H. H.

Chemistry of New Zealand *Melicope* Species. VII—Relation between the Constitution of Some Flavonol Derivatives and their Acidity, Basicity, Colours with Ferric Chloride, and Ultraviolet Absorption Spectra. L. H. Briggs and R. H. Locker. *J.C.S.*, 3136–3142 (Nov. 1951).

The relation between the constitution of 18 related flavonol derivatives and the properties listed in the title is discussed. H. H. H.

Dimethylpteridine Red. P. Karrer and H. Feigl. *Helv. Chim. Acta*, **34**, 2155–2158 (Dec. 1951).

From 2-amino-6-hydroxy-8-methylpteridine and 2-amino-6-hydroxy-8:9-dimethylpteridine, dimethylpteridine red (I) was obtained by oxidation with gaseous oxygen. Ozonolysis leads to 8-methylisoxanthopteridine and 2-amino-6-hydroxy-8-methylpteridine-9-carboxylic acid.



(I)

G. L.

Dichroism of Planar Complexes. III—Colour and Structure of the Crystals of Tetracyanoplatinates (II). S. Yamada. *Bull. Chem. Soc. Japan*, **24**, 125–127 (July 1951).

The dichroism of crystals of $BaPt(CN)_4.4H_2O$, $CaPt(CN)_4.5H_2O$, and $MgPt(CN)_4.7H_2O$ has been measured in the visible and near ultraviolet by means of a microscopic method using microcrystals. These crystals are coloured although their solutions are colourless, and they show similar dichroism with the characteristic sharp absorption band for parallel absorption. It is found that the band becomes sharper and displaced towards the longer wavelengths in the order of Ca, Ba, and Mg salts. The production of this absorption band is associated with direct interaction between the tetracyanoplatinate ions, the band becoming sharper and displaced to longer wavelengths as the interaction becomes greater. This is due to the decreased distance between these ions in the order of the Ca, Ba, and Mg salts. W. J. M.

PATENTS

Preparation of Diazo Compounds. N.V. Philips' Gloeilampenfabrieken. *BP* 659,897

Aromatic aminosulphonic acids of low solubility in water may be very successfully diazotised by suspending them in water containing a nitrite, cooling, and adding a little strong acid. Provided that the diazo compound so produced is a stronger acid than HNO_2, the latter will be steadily liberated and the reaction will proceed smoothly to completion. The amount of strong acid needed to initiate the reaction is 0·005–0·02 mol.; HNO_3 is very suitable, or fluoroboric acid if the diazo compounds are intended for use in photographic work. Thus 2-amino-6-methylphenol-4-sulphonic acid (150 g.) is stirred into water (400 c.c.) containing $NaNO_2$ (60 g.). At < 10°c. fluoroboric acid (3 c.c. of ca. 90%) is added in drops. The reaction takes about 10 min., the temperature rising to 40–50°c. By cooling to 0°c., sodium 2-diazo-6-methylphenol-4-sulphonate (140 g.) can be isolated. Similarly, the silver salt of the same diazo compound can be isolated by using $AgNO_2$ as the source of HNO_2. E. S.

Azoic Diazo Components—Improvements in Filtering and Dusting Properties. General Aniline. *BP* 661,533

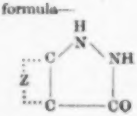
By preparing stable double salts of diazonium compounds with metallic chlorides, fluoroborates, naphthalene-1:5-disulphonic acids, etc. in presence of anionic surface-active agents, better filtration characteristics of the products are ensured, and the resulting dried powders are frequently much less dusty. About 5–20% of the surface-active agent, based on the amount of diazonium compound, is employed; alkali-metal soaps, sulphonated castor oil, sulphonated and sulphated higher fatty alcohols, and acylaminoalkylsulphonates and similar products all give good results. Thus, all parts being by weight, 2-amino-4-nitroanisole (33·6) is diazotised, and the solution filtered and mixed with a solution of sodium oleate (6). This solution is run into a solution of naphthalene-1:5-disulphonic acid (57·6), and the precipitated double salt filtered off and dry-mixed to standard strength with Na_2SO_4, $Al_2(SO_4)_3$, and $MgSO_4$. Owing to the coarser crystalline structure of the precipitated stabilised diazo compound compared with the product obtained without the addition of sodium oleate, a drier filter cake is produced and the final powder is less dusty. E. S.

Diazotisation of Aromatic Aminosulphonic Acids in Presence of Ketones. N.V. Philips' Gloeilampenfabrieken. *BP* 660,895

Smooth and rapid diazotisation of many aromatic aminosulphonic acids can be achieved by conducting the reaction in presence of one or more water-soluble aliphatic ketones. Acetone, at concentrations of 20–75%, is the preferred ketone. In many cases, the diazo compound can be separated by adding more ketone at the completion of the reaction. Thus 1-amino-2-naphthol-4-sulphonic acid (23·9 g.) is suspended in a mixture of water (30 c.c.), acetone (20 c.c.), and conc. HCl (20 c.c.), and cooled to 5°c. Whilst stirring, a solution of sodium nitrite (8·5 g.) in water (20 c.c.) is slowly added, the temperature rising to about 15°c. After about 15 min., more acetone (30 c.c.) is added, whereupon sodium 1-diazo-2-naphthol-4-sulphonate crystallises. E. S.

Photographic Colour Couplers. Gevaert. *BP* 663,190

Compounds of formula—



(Z = atoms to complete an iso- or hetero-cyclic nucleus or a polycyclic nucleus) form magenta dyes with mono-, di-, or tri-aminoaryl compounds in presence of a reducible silver salt. C. O. C.

Colour Couplers. Gevaert. *BP* 663,564

Polymeric compounds or polycondensates containing 10 mol. of one or more dye intermediates per kg., these intermediates being of formula—

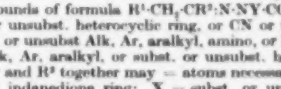
(R = H or a substituent which splits off during development; Z = an organic radical; X = an electronegative radical) and attached to the polymer or polycondensate by the free bond, are valuable colour couplers for use in conjunction with aromatic amino developers. C. O. C.

Acylhydrazones as Colour Couplers. Gevaert. *BP* 663,550

Compounds of formula $R^1{\cdot}CH_2{\cdot}CR^2{:}N{\cdot}NY{\cdot}CO{\cdot}X$ (R^1 = subst. or unsubst. heterocyclic ring, or CN or CO, linked to subst. or unsubst Alk, Ar, aralkyl, amino, or hydrazino; R^2 = Alk, Ar, aralkyl, or subst. or unsubst. heterocyclic ring; R^1 and R^2 together may = atoms necessary to complete an indanedione ring; X = subst. or unsubst. Ar, aralkyl, or heterocyclic; Y = H or subst. or unsubst. Alk, Ar, aralkyl, or heterocyclic) act as colour developers in conjunction with primary aromatic developing agents. C. O. C.

Colour Couplers. Kodak. *BP* 662,316

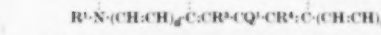
Compounds of formula—



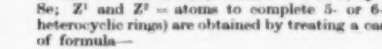
(R = subst. or unsubst. phenyl or α- or β-naphthyl; X = a carboxylic acyl group) yield magentas with primary amino developing agents in presence of exposed silver halide emulsions. C. O. C.

Bisacetone Compounds and Carbocyanine Dyes therefrom. Kodak. *BP* 663,440

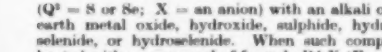
Compounds of formula—



(R^1 and R^2 = Alk or may = Ar when Z = non-metallic atoms to complete a heterocyclic nucleus of the thiazole, benzothiazole, thiazoline, or 3:3-dialkylindolenine series; R^3 and R^4 = H or Alk; d and s = 0 or 1; Q^1 = O, S, or Se; Z^1 and Z^2 = atoms to complete 5- or 6-membered heterocyclic rings) are obtained by treating a carbocyanine of formula—



(Q^2 = S or Se; X = an anion) with an alkali or alkaline-earth metal oxide, hydroxide, sulphide, hydrosulphide, selenide, or hydroselenide. When such compounds are heated with a compound of formula $R^5{\cdot}X$ (X = an anion; R^5 = an organic group) they yield carbocyanine dyes of formula—



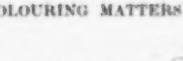
C. O. C.

Polymeric Acetals as Colour Formers. DuP. *BP* 662,793

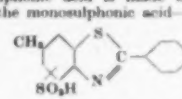
Acetals of *m*-3-methylsalicylamidobenzaldehyde with water-soluble or hydrophilic polymers containing several recurring intralinear $-CH_2{\cdot}CH(OH)-$ groups are useful as photographic colour formers. C. O. C.

Yellow, Substantive Monoazo Dyes. Ciba. *BP* 659,130

Greenish-yellow dyes for cotton are made by diazotising dehydrothio-*p*-toluidinedisulphonic acid and coupling with an acetoacetarylide—



(X = Cl or Br; Alk = CH_3 or C_2H_5). The dehydrothio-*p*-toluidinedisulphonic acid is made by baking the acid sulphate of the monosulphonic acid—



whereupon a second sulpho group is introduced *ortho* to the NH_2 group. The dyes produced have better affinity and wet fastness than the dyes of *BP* 6644 (1914) in which acetoacetyl-*o*-anisidide, -*p*-anisidide, -*o*-chloroanilide, and -*p*-chloroanilide are coupled with this diazo component. E. S.

Brown, Metachrome, Monoazo Dyes. S. *BP* 658,871

Brown, chromable dyes, many of them applicable by the metachrome process, are made by diazotising a 6-acylamino-2-amino-4-nitrophenol, and coupling with a 2-acylamino-4-alkyl(or aryl or *cyclo*alkyl)phenol. Thus 2-amino-6-benzoylamino-4-nitrophenol → 2-acetylamino-4-methylphenol dyes wool red-brown converted to dark brown by afterchroming, and may also be dyed metachrome. E. S.

Metachrome Monoazo Dyes. Ciba. *BP* 659,695

Mainly navy blue and black metachrome dyes are made by coupling a diazotised *o*-aminophenol with a 2-naphthol-6-sulphonamide. The dyes produced contain no carboxyl or sulpho groups, but the *o*-aminophenol may carry substituents, especially nitro, and the amide group may carry alkyl or hydroxyalkyl substituents. Thus 2-amino-4-chloro-5-nitrophenol → 2-naphthol-6-sulphonamide is a navy blue metachrome dye. E. S.

Monoazo Acid Dyes containing an *N*-Alkylsulphamyl Group. Ciba. *BP* 660,196

Bright orange and red acid dyes are made by diazotising a primary amine of the benzene series, which contains an *N*-alkylsulphamyl group $-SO_2{\cdot}NH{\cdot}Alk$ (Alk of 8–12 C), and coupling with a naphtholsulphonic acid. Thus 2-aminoanisole-4-sulphon-*n*-dodecylamide → G acid dyes wool pure orange from an acetic acid bath. E. S.

Orange and Brown Azo Dyes for Chrome Printing on Cellulose Fibres and for Dyeing Anodised Aluminium. DH. *BP* 660,079

Mordant dyes which give oranges and browns by chrome printing on cellulose fibres, and also dye anodised aluminium, are made by condensing a nitromonoazo dye $A^1{\cdot}N{:}N{\cdot}B^1{\cdot}NO_2$ in an alkaline medium with an aminodiaryl ketone $A^2{\cdot}CO{\cdot}B^2{\cdot}NH_2$ (A^1, A^2 = *o*-hydroxycarboxyaryl residues; B^1, B^2 = arylene). Thus, all parts being by weight, 3′-carboxy-4′-hydroxy-4-nitroazobenzene-3-sulphonic acid (36·7) and 5-*p*-aminobenzoylsalicylic acid (25·7) are heated with 80% NaOH (400) for 15 hr. under reflux. The product gives oranges when chrome-printed on cotton, and dyes anodised aluminium golden tints. E. S.

Metalliferous Azo Dyes. Basf. *BP* 660,447

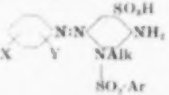
Copper complexes of *oo′*-dihydroxyazo dyes can be made by treating *o*-monohydroxyazo compounds, having a free *o′*-position, with copper salts, in presence of suitable oxidising agents (H_2O_2, alkali-metal peroxides, perborates, and percarbonates, organic peracids and acyl peroxides, or chromic acid; anodic oxidation may also be used) under slightly acid conditions. The copper so introduced may be removed from the molecule by treatment with strong mineral acid or alkali-metal sulphides to yield the *oo′*-dihydroxy body, which may then be remetallised, e.g. with chromium, in substance or on the fibre. Thus *p*-nitroaniline → resorcinol ← metanilic acid is treated at 50° c. with $CuSO_4$ and H_2O_2 to give a brown leather dye. Or the monoazo compound naphthionic acid → 1-naphthol-5-sulphonic acid is treated with $CuSO_4$ and H_2O_2. The copper complex so formed is isolated, and decoppered by treatment with e.g. strong HCl. By chroming the *oo′*-dihydroxyazo compound so produced, a blue wool dye is formed, identical with the product of chroming 1-amino-2-naphthol-4-sulphonic acid → 1-naphthol-5-sulphonic acid. E. S.

Chromable Monoazo Dye for Wool. General Aniline. *BP* 661,819

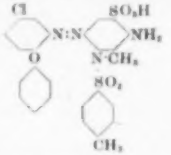
The monoazo compound 2-amino-6-nitrophenol-4-sulphonic acid → 1-*o*-carboxylbenzoylamino-7-naphthol is a bright green metachrome dye; the hue is little affected by artificial light. Dyed afterchrome it is olive-black and leaves acetate rayon effect fibres unstained. E. S.

Disazo Acid Dyes. S. *BP* 660,056

Violet, blue, and black acid dyes are made by diazotising an aminoazo compound—



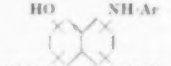
(X = Alk of 1–4 C, O-Ar, O-Aralkyl, NAlk·SO_2·Ar, NAralkyl·SO_2·Ar, SO_2·NAlk·Ar, SO_2·NAralkyl·*cyclo*Hexyl, or SO_2·NAr·Aralkyl; Y = H, Hal, or Alk) and coupling under acid conditions with γ acid or its *N*-alkyl or *N*-aryl derivatives. The dyes so produced have better milling fastness, better exhaustion from neutral dyebaths, and in some cases better artificial light appearance than the similar dyes of *GP* 695,401 which contain no sulphonamido groups. Thus the aminoazo dye 2-amino-4-chlorodiphenyl ether → 3 - *p* - tolylaulphonylaminoaniline - 6 - sulphonic acid is treated with dimethyl sulphate to form the *N*-methyl compound—



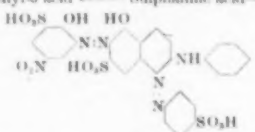
This is diazotised and coupled with γ acid in presence of acetic acid to yield a blue acid dye. E. S.

Azo Dyes containing an Azine Ring. S. *BP* 659,467

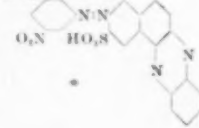
Azo dyes containing an azine ring are obtained when azo dyes—



(the naphthalene nucleus contains at least one SO_3H; the Ar has a free position *ortho* to the NH; R^1 and R^2 may contain further azo linkages; and N:N·R^1 and NH·Ar, as well as N:N·R^2 and OH, are in the *ortho* position to one another) are treated with acids or acid salts. Higher yields are obtained by thus making the azine ring closure the final operation, as the known hydroxybenzophenazinesulphonic acids or hydroxydinaphthopyrazinesulphonic acids do not couple readily with diazo compounds. Thus the disazo dye—2-Amino-4-nitrophenol-6-sulphonic acid $\xrightarrow{\text{Alkali}}$ → Phenyl-J acid ← $\xrightarrow{\text{Acid}}$ Sulphanilic acid—



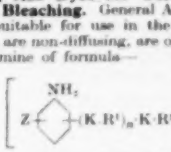
is treated at 85–90°c. with dil. HCl, when sulphanilic acid splits off, and the azine ring is formed, the product being—



It dyes wool brownish red, converted to fast olive by afterchroming. E. S.

Non-diffusing Azo Dyes for Use in Photographic Azo Dye Bleaching. General Aniline. *BP* 663,005

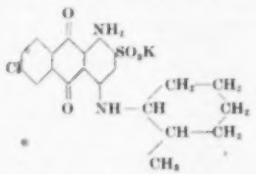
Azo dyes, suitable for use in the azo dye bleaching process, which are non-diffusing, are obtained by coupling a diazotised amine of formula—



(Z = an aliphatic substituent or Hal; K = a homopolar linkage, e.g. a peptide NH·CO linkage; R^1 = an aromatic radical; R^2 = an aliphatic chain of > 7 C; *n* = 0 or 1; *p* = 1 or 2), e.g. tetradecyloxyaniline, with suitable couplers, e.g. naphthols for magentas and pyrazolones for yellows. Cyan dyes are obtained by coupling with e.g. naphthylamine, diazotising the product, and then coupling with α-naphthol. C. O. C.

Anthraquinonoid Acid Dyes. S. *BP* 657,262

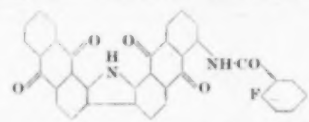
Blue acid dyes faster to fulling than those of *BP* 276,408 (*J.S.D.C.*, **44**, 28 (1928)) are made by condensing 1-amino-4-bromo(or chloro)anthraquinone-2-sulphonic acid containing chlorine or bromine in the 5, 6, 7, or 8 position with a *cyclo*hexylamine substituted with one or more Alk or Ar. Alternatively the corresponding 2:4-dihalogenoanthraquinone may be used, and the 2-halogen replaced later by SO_3H by treating with a sulphite. Thus the dye—



is prepared by heating together at 65°c. for 16 hr. potassium 1-amino-4-bromo-6-chloroanthraquinone-2-sulphonate, 2-methyl*cyclo*hexylamine, copper powder, and aqueous-alcoholic NaOH. R. K. F.

Carbazole Vat Dyes containing *o*-Fluorobenzoylamino Groups. Ciba. *BP* 658,721

m'-Dianthrimides having at least one further *a*-position substituted with an *o*-fluorobenzoylamino group, and prepared e.g. by condensing a 1-aminoanthraquinone with a 1-chloroanthraquinone, one or both of which contain an *o*-fluorobenzoylamino group, are cyclised to the corresponding carbazole derivatives. Thus the yellow dye—



is prepared by first heating 1-chloro-5-*o*-fluorobenzoylaminoanthraquinone, 1-aminoanthraquinone, Na_2CO_3, and copper acetate in nitrobenzene at 200–205°c. for 16 hr. The resulting dianthrimide is cyclised by stirring for 2 hr. at 20°c. with $AlCl_3$ and *o*-fluorobenzoyl chloride in nitrobenzene. R. K. F.

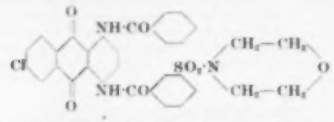
o-Fluorobenzoylaminoanthraquinones — Vat Dyes. Ciba. *BP* 658,722

1:5-Diaminoanthraquinone is acylated so that at least one *o*-fluorobenzoylamino group is present in the resulting yellow vat dye. Thus 1-amino-5-*o*-fluorobenzoylaminoanthraquinone, prepared by heating 1:5-diaminoanthraquinone and *o*-fluorobenzoyl chloride at 200–205°c. in nitrobenzene, is heated at 145°c. in nitrobenzene with oxalyl chloride. R. K. F.

1 : 4 - Diacylaminoanthraquinone-Sulphonamides — Vat Dyes. Ciba. *BP* 656,870

1-Acylamino-4-aminoanthraquinones are acylated with a *m*-dialkylsulphonamide or *m*-dialkylenesulphonamide of

benzoic acid, or a functional derivative thereof, to yield pink vat dyes. Thus the dye—



is prepared by heating together the *m*-sulphonmorpholide of benzoic acid, 4-amino-1-benzoylamino-6-chloro-anthraquinone, and thionyl chloride in nitrobenzene for 2 hr. at 125–130°c. R. K. F.

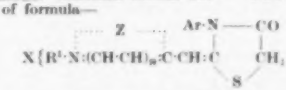
Azine Dyes. General Aniline. *USP* 2,555,127

Azine dyes are formed by developing an exposed silver halide emulsion in presence of a colour former with a compound of formula—

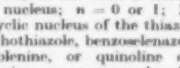
(R = an aliphatic or aromatic radical; X = sulpho or carboxy). C. O. C.

Methin Dyes containing a 3-Aryl-4-thiazolidine Nucleus. Eastman Kodak Co. *USP* 2,556,515

Dyes of formula—



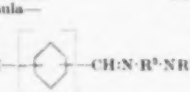
(X = an anion; R^1 = Alk or Ar when Z forms a benzothiazole or thiazoline nucleus; n = 0 or 1; Z = atoms to complete a heterocyclic nucleus of the thiazole, benzothiazole, α- or β-naphthothiazole, benzoselenazole, thiazoline, 3:3-dimethylindolenine, or quinoline series) are obtained by condensing a compound of formula Hal·CH_2·CO·O·R^2 (R^2 = Alk or Ar) with one of formula—



C. O. C.

Azomethin Dyes. Kodak. *BP* 663,114

Compounds of formula—



(n = 0 or 1; R^1 and R^2 = H or subst. or unsubst. Alk; R^3 = subst. or unsubst. *p*-phenylene group) are photographic filter dyes which are very readily bleached, the decomposition products being readily removed during normal developing and fixing. C. O. C.

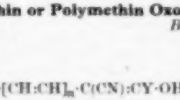
***mero*Cyanine Dyes and Intermediates.** Ilford. *BP* 662,775–6

Compounds of formula—

(R = Alk or aralkyl) form *mero*cyanine dyes with any intermediate which forms *mero*cyanine dyes with *N*-subst. rhodanines. C. O. C.

Unsymmetrical Methin or Polymethin Oxonol Dyes. Kodak. *BP* 663,042

Dyes of formula—



(X = atoms to complete a nucleus of barbituric or thiobarbituric acid, isoxazolone, indanedione, pyrazolone, or oxindole; n = 0–2; Y = benzene or benzofuran nucleus) are good filter and antihalation dyes. C. O. C.

Carbon Black. Godfrey L. Cabot. *BP* 663,237

Carbon black is rendered more suitable for compounding with rubber by treating it to increase its volatile content and then reducing the volatile content to that it originally had. C. O. C.

Carbon Black. Phillips Petroleum Co. *USP* 2,556,196

A process for gaining increased yields of furnace black. C. O. C.

Mixture of Phloroglucinol or Resorcinol and α-Naphthol as Diazotype Developer (IX p. 103).

Yellow Dye Images (IX p. 103).

V— PAINTS; ENAMELS; INKS

Physical Chemistry of Protective Coatings. H. Lomax. *Paint, Oil & Chem. Rev.*, **114**, (17), 15–16, 18 (1951): *Chem. Abs.*, **45**, 9891 (10 Nov. 1951).

Oil absorption is governed by the pigment density, range of particle sizes, and the degree of pigment flocculation. Stability is affected by pigment settling, physico-chemical changes at the interfaces leading to breakdown of emulsions or dewetting of pigments and resulting in hard settling, or chemical changes resulting from reaction of the vehicle with reactive pigment. Brushing and flow properties are a function of the structure of vehicle and pigment, or of the thixotropic properties of the paint. C. O. C.

Dyes for the Surface Staining of Wood. J. K. Fishpool. *Paint Technol.*, **16**, 245–249 (1951): *Chem. Abs.*, **45**, 9889 (10 Nov. 1951).

Based on the vehicle used there are five main classes of stains—water, oil, wax, spirit, and non-grain-raising stains. Water-soluble acid dyes are fairly fast to light but are weak and dull compared with water-soluble basic dyes. Addition of 1–2% of NH_4OH or 0·5% of a wetting agent to the aq. soln. of acid dye results in better penetration. Water-soluble basic dyes are deficient in fastness to light and "bleed through" when covered with spirit varnish, but are used where outstanding brightness is needed. Vat dyes are used where maximum fastness to light is required; they are applied as leuco compounds. Spirit stains are the most widely used to-day. They are usually solutions of dyes in methanol and have rapid and deep penetration and fastness to rubbing and water. Such dyes as the amine salt of Cu phthalocyaninetrisulphonic acid and the *cyclo*hexamine salt of Anthraquinone Violet yield spirit stains of good fastness to light. Oil stains are dyes dissolved in aromatic hydrocarbon solvents or varnish. Non-grain-raising stains are fast-to-light dyes dissolved in mixtures of glycollic solvents, hydrocarbons, and/or methanol. The glycol, being the last to evaporate, retains the dye long enough in solution to stain the wood evenly and deeply. They are relatively clear, and may bleed when coated with lacquers containing Cellosolve or similar solvents. Dyes for stains are usually tested for hue, strength, light-fastness, and bleeding into lacquer. Suitable tests are described. C. O. C.

PATENTS

Steam-setting Printing Ink. Sun Chemical Corpn. *BP* 663,918

An ink composition which rapidly sets upon application of heat or steam, but which is nevertheless readily removed from the printing press by use of aqueous wash-up materials, comprises as binder a water-immiscible resin, as carrier a water-immiscible liquid glycol or polyglycol, or an ester or ether thereof, and a water-dispersible detergent mixture containing a hydroxyalkylamine soap of a higher fatty acid, a hydroxyalkylamine, and a non-ionic surface-active agent. C. O. C.

Cellulose Propionate Lacquers. Celanese Corpn. of America. *BP* 661,639

Lacquers useful for protecting metal surfaces from corrosion comprise cellulose propionate (15–40 parts) in a medium (100) consisting of toluene or xylene and propyl, *iso*propyl, butyl, or an amyl acetate (10–50) in such proportions that the composition is a gel at atmospheric temperature and is capable of reversible change to a liquid on heating and of irreversible change to a coherent film on evaporation of the medium. W. G. C.

VI—FIBRES; YARNS; FABRICS

Action of Alkali and Oxygen on Cellulose. A. Schaeffer. *Textil-Rund.*, **6**, 489–498 (Nov. 1951).

Previous work on the influence of alkali boiling on cellulose fibres (*ibid.*, **6**, 159 (March 1951)) has been extended in order to determine the influence of oxygen which may be contained in the alkaline liquor. Rayon staple and cotton fibres were boiled fifty times in alkaline liquors (1) with a current of air passed through the liquor, (2) in a liquor freed from oxygen in an atmosphere of nitrogen. Measurements of fibre damage showed that the presence of oxygen caused increased damage only with cuprammonium rayon and not with viscose rayon or cotton. This was thought to be due to the presence of traces of copper in the cuprammonium rayon. Support for this view was obtained when viscose rayon was found to suffer increased degradation as a result of adding copper salts to the alkaline liquor. Iron salts and street dust had a similar catalytic action. Addition of reducing agents decreased degradation due to alkali boiling, an addition of pyrocatechol being particularly effective. A possible series of reactions to explain the degradation would be hydrolysis by alkali of glucopyranose rings, followed by oxidation of the resulting CH(OH) group in the 1-position to a CO group. This would give an ester linkage, which when hydrolysed would cause splitting of the cellulose chains. This mechanism would explain the observed increase in carboxyl groups and absence of keto groups in the product. Tests with Luminol indicated that hydrogen peroxide is formed during the reaction. A series of alkali-boiling treatments was therefore carried out in the presence of stabilisers for hydrogen peroxide. Degradation was decreased by addition of magnesium silicate, but silicates were deposited on the fibres, which were rendered harsh. Other stabilising agents, particularly Trilon B, were effective without giving this difficulty. F. A.

Apparent Mildew Resistance of Weathered Cotton Duck. E. Abrams. *Text. Research J.*, **21**, 714–720 (Oct. 1951).

The observation that untreated cotton fabric which had been exposed to sunlight would no longer support mildew growth to the same extent as the original fabric has been re-examined. It is shown that the fabrics exhibited a degree of resistance to *Chaetomium globosum* which increased up to the fourth month of outdoor exposure, after which the resistance remained essentially constant until the eighth month, the longest time examined. A possible explanation is that the deterioration of the "amorphous" cellulose is accomplished most rapidly by photochemical action, leaving essentially the "crystalline" component of the cotton, which is more highly resistant to *C. globosum*. A. B.

PATENTS

Pigmented Solutions of Cellulose Esters or Ethers. Courtaulds. *BP* 664,149

Dry powdered cellulose ester or ether and not $> 12\%$ of its weight of a dry pigment are mixed until the pigment is uniformly distributed among the ester or ether particles. The mixture is then stirred with a solvent until a homogeneous solution is obtained, which is suitable for spinning into filaments. C. O. C.

Continuous Spinning of Viscose Rayon. Courtaulds. *BP* 661,939

In continuous spinning the time of treatment is reduced by passing the incompletely regenerated viscose to a thread-advancing reel maintained at $> 75°$C. and spraying with an aqueous acid liquid at $< 75°$C., so that heat is supplied from the reel to the regenerating liquid.

BP 661,940

Another method of increasing the speed of regenerating viscose is to pass the thread through an acid zone and then into an aqueous oxidising zone, e.g. aqueous sodium hypochlorite. W. G. C.

Permanently Set Viscose Rayon. Comptoir des Textiles Artificiels. *BP* 661,476

Yarn having a strong set curl, which is permanent under normal conditions of use, is made by extruding viscose into a bath containing aluminium sulphate and an alkali-metal bisulphate, e.g. sodium bisulphate, passing the yarn through a hot aqueous bath whilst stretching $> 50\%$, washing the yarn, and drying without tension. W. G. C.

Collagen Threads and Filaments. CYC. *BP* 663,032

Collagen solution is extruded into a bath of e.g. ammonium sulphate of pH 7·8 at 20–30°c., collected, stretched, pretanned in chromic sulphate–ammonium sulphate of pH 4·8 for 24 hr., softened, stretched, tanned, rinsed, and dried. Such material is strong, uniform, easily sterilised, and therefore useful for surgical sutures. J. W. B.

VII—DESIZING; SCOURING; CARBONISING; BLEACHING

PATENTS

Bleaching of Bast Fibres. ICI. *BP* 662,890

A good bleach is obtained by treating with a hypochlorite soln. initially at pH not ≥ 8 and finally at pH > 8 and then with a hot solution of an alkali or alkaline-earth metal chlorite. Intermediate washing is unnecessary. C. O. C.

Action of Alkali and Oxygen on Cellulose (VI this page).
Determination of the "Whiteness" of Textile Materials (XIV p. 108).

VIII—DYEING

Microscopical Dyeing Phenomena; Studies with the Microdyeoscope. H. E. Millson and L. H. Turl. *Text. Research J.*, **21**, 685–702 (Oct. 1951).

An instrument called the "Microdyeoscope" is described, which permits both visual and photographic observation of dyeing phenomena under conditions approximating to the usual commercial dyeing processes. It permits continuous microscopic examination of the dyebath and fibre, and provides means for control of circulation, temperature, and dyebath additions and for rinsing and repeated cyclic wet treatments. The capacity of the dyeing chamber is 330 ml. Applications of the Microdyeoscope detailed include— the dyeing of wool with acid- and neutral-dyeing wool dyes, the mechanism of acetate rayon dyeing, precipitation in the dyebath, bronzing of direct cotton dyes, precipitation in top-chrome and metachrome dyeings, levelling agents in wool dyeing, and histological studies on the wool fibre. A. B.

Effect of Agitation on Dyeing. I—Wool and Acid Dyes. A.A.T.C.C. Northern New England Section. *Amer. Dyestuff Rep.*, **40**, P 851–P 852, P 866 (24 Dec. 1951).

Dyeings of Erio Phloxine 2G (*C.I.* 31) are made on wool at 60 ± 1°c., under conditions of standard though variable agitation. Results indicate that manner of agitation is as important as the amount, and also that increased agitation reduces the concentration of dye at the fibre surface. J. W. B.

Fading of a Dye on Cellulose Acetate by Light and by Gas Fumes—1 : 4-Bismethylaminoanthraquinone. M. Couper. *Text. Research J.*, **21**, 720–725 (Oct. 1951).

A study is made of the "fading products" isolated from faded dyeings of 1:4-bismethylaminoanthraquinone on cellulose acetate, nylon, and acetylated nylon. Chromatographic analysis showed that these products included each of the six possible 1:4-disubstituted anthraquinones containing methylamino, amino, or hydroxy groups. There was also evidence of other anthraquinone nuclei. The products of severe gas-fume fading on cellulose acetate bear a striking resemblance to those from light fading. It has also been demonstrated that the presence of primary amino groups in nylon is not the explanation for the freedom from gas-fume fading on this fibre. A. B.

Monte-Carlo Method for Solving Diffusion Problems. W. King. *Ind. Eng. Chem.*, **43**, 2475–2478 (Nov. 1951).

A new means of solving problems in physics and engineering by means of large-scale computing machines is described. A realistic model of situations involving classical physics or chemistry can be inserted into a computer, the actual calculations used being extremely elementary, consisting of many additions of unity in certain counters chosen by random numbers. The ideas of the method are illustrated by consideration of the problem of distribution of a dye supplied to the centre of a capillary

tube and diffusing in both directions. The model can be set up for three-dimensional problems just as easily as for one, and other features of the physical situation such as special boundary conditions, which often cause trouble in solving differential equations, can be given to the machine in a simple way. Changes of composition by chemical reaction may take place during diffusion. A diffusion problem in connection with the statistical mechanics of polymers is discussed; this is an attempt to find certain properties of random configurations of long-chain molecules. A. H.

PATENTS

Dyeing Cellulose Esters or Ethers with Vat Dyes. Celanese Corpn. of America. *BP* 664,340

The material is impregnated with a finely divided vat dye, and then treated with an aqueous reducing liquor made alkaline with NaOH and containing a water-miscible organic swelling agent for the cellulose ester or ether; this is followed by reoxidation of the dye. Reduction can be effected in 15–90 sec., so that the material need be swollen only for a very short period. C. O. C.

Dyeing Spun Cellulose-derivative Staple Fibres in Alcoholic Solution and then Skying. Celanese Corpn. of America. *USP* 2,552,807

Acetate rayon or other organic derivative of cellulose is dyed rapidly and level by pad dyeing or the like using a liquor consisting solely of dye, a lower aliphatic alcohol, and 0–5% of water. The impregnated fabrics are dried by skying the material over rolls. The dried fabric does not require scouring to remove excess dye, a simple rinse in water being sufficient. Penetration is excellent, and greatly increased colour value is obtained. C. O. C.

Preventing Formation of Sludge when Vat or Sulphur Dyeing by the Molten Metal Process. Standfast Dyers & Printers. *BP* 663,066

Modification of *BP* 620,584 (*J.S.D.C.*, **65**, 412 (1949)). Formation of sludge on the surface of the molten metal is prevented by adding an antioxidant, e.g. glucose, benzaldehyde, gallic or tannic acid, to the impregnating vat. C. O. C.

Pigment Dyeing. IC. *BP* 663,824

Modification of *BP* 661,085 (*J.S.D.C.*, **68**, 75 (Feb. 1952)). When dyeing with the very low concentration of pigment and binder necessary to preserve the appearance and porosity of textiles, flocculation of the pigment is avoided by dispersing it in the aqueous phase instead of the lacquer phase. Preferably the aqueous phase contains a water-soluble bodying agent, e.g. polyvinyl alcohol, a water-soluble phenol–aldehyde or melamine–formaldehyde resin, or sucrose octa-acetate. C. O. C.

Colouring Textiles. Copeman Laboratories. *BP* 663,853

The material is impregnated with an aqueous emulsion of a solution in a water-insoluble organic solvent of a water-insoluble resin, a water-insoluble dye, and an emulsifying agent, and then dried. C. O. C.

Controlling the pH of Wool Dyebaths containing Boric Acid. W. E. Saxby (Nottingham). *BP* 662,795

Modification of *BP* 638,176 (*J.S.D.C.*, **66**, 498 (1950)). The pH of the dyebath is controlled by use of an alkaline salt of pyro- or meta-boric acid with or without free boric acid. C. O. C.

Gas-fading Inhibitor (III p. 96).

NN'N''-Triphenylguanidine as Gas-fading Inhibitor (III p. 96).

IX—PRINTING

PATENTS

Printing Paste. Monsanto. *BP* 663,769

A paste consisting of a dye and as thickener an aq. soln. or dispersion of an ammonium, amine, or alkali-metal salt of a copolymer of styrene or a nuclear-substituted styrene with maleic anhydride or incompletely esterified maleic anhydride, which copolymer is cross-linked with divinylbenzene, is used. Only small amounts of the thickener are required, and the pastes give excellent colour yield and are readily removed from the fabric. Alternatively if, in addition to divinylbenzene, the copolymer is also cross-linked with formaldehyde, then after steaming and ageing to develop the print, baking at 260–300°F. sets the paste in the fabric, so giving a permanent finish. C. O. C.

Screen Printing. M. S. Striker. *BP* 660,261

Fastening the cloth to be printed on to the table by a slightly sticky adhesive applied to the table and remaining thereon in a permanently slightly sticky condition after the cloth has been printed and removed enables successive lengths of the cloth to be printed without renewal of the adhesive. C. O. C.

Screen Printing Ink. DuP. *USP* 2,556,241

A composition containing pigment (60–80% of the non-volatile components), a soluble vinyl resin, a solvent, and a plasticiser is suitable for screen printing on vinyl-resin-coated fabrics. It does not cause the screen to stick to the fabric, nor are "legs" or stringiness formed when the stencil is being pulled away from the fabric. C. O. C.

Colour Prints from Colour Negatives or Positives. Kodak. *BP* 662,789

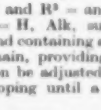
Modification of *BP* 660,099 (*J.S.D.C.*, **68**, 41 (Jan. 1952)). The integrated light used consists of the whole of the printing light transmitted by the negative together with some light direct from the source, the "diluting" light being excluded from the transmitted printing light beam when printing on the light-sensitive material. This enables good prints to be obtained even from abnormal negatives or positives. C. O. C.

Mixture of Phloroglucinol or Resorcinol and α-Naphthol as Diazotype Developer. Spolek pro Chemickou a Hutní Výrobu. *BP* 663,575

A mixture of phloroglucinol or resorcinol and α-naphthol used as a diazotype developer yields homogeneous deep colour prints without any smearing. C. O. C.

Yellow Dye Images. General Aniline. *BP* 662,419

Yellow images can be produced simultaneously with cyan and magenta azine dye images by development with a compound of formula—



(R^1 = H or Alk; R^2 and R^3 = an aliphatic or aromatic radical; X and Y = H, Alk, sulpho, or carboxy) in presence of a compound containing an activated methylene group in an open chain, providing that the pH of the photographic emulsion be adjusted with an alkali in the final phase of developing until a yellow image clearly manifests itself. C. O. C.

Mechanical Application of Transfers to Ceramics. William Boulton. *BP* 664,045

X—SIZING AND FINISHING

Infrared Radiant Drying of Textiles. F. M. Tiller and W. K. Lantner. *Amer. Dyestuff Rep.*, **40**, P 854–P 866 (24 Dec. 1951).

Experimental runs in ovens equipped with infrared lamps were made both with small sections of cloth in stationary tests and with continuous passage of 18 in. wide cloth between two banks of lamps. Small-scale tests were made to study the effect of colour, moisture content, and weight on radiation absorption; in one example a maximum efficiency of 50–55% for a white cloth compares with 70–80% for a dark material, which absorbs more radiation. Rates of drying of 7 lb./hr./sq. ft. were obtained in a continuous drier. J. W. B.

Incorporation of Acrylate Resins into Regenerated Cellulose Fibres. A. Nowakowski. *Text. Research J.*, **21**, 740–746 (Oct. 1951).

The utility of the acrylate resins in the aftertreatment of regenerated cellulose fibres is discussed. Various methods of application were investigated, but the physical properties of the fibres are unchanged, although the resin (up to 10-5% uptake) is distributed evenly throughout the fibre. It is possible that an aftertreatment such as cold-drawing may cause an improvement in the properties. A. B.

Abrasion Resistance of Resin-treated Fabrics. A. C. Nuessle. *Text. Research J.*, **21**, 747–749 (Oct. 1951).

Graphs show the abrasion resistance of resin-treated fabrics as a function of the pressure on the abrading

surface. An analysis of the scatter of points shows that at low load a resin-treated fabric, having higher elastic recovery, will withstand abrasion better. At higher resin concentrations the fabric has less resistance to severe stresses, presumably because of the reduction in elongation. A. B.

PATENTS

Non-woven Fabrics from Mixtures containing Potentially Adhesive Fibres. American Viscose Corpn. *BP* 664,100

A fibrous product comprises pressed slivers or rovings containing potentially adhesive fibres, inter-sliver adhesion being effected by protruding fibres. In one example a number of rovings containing 10% of fibrous vinyl chloride–vinyl acetate copolymer are passed side by side between heated calender rolls at about 365° F. and 25 lb./sq. in., with a pressing time of 10 sec. A thin, strong felt-like porous material, highly flexible transversely, is produced. J. W. B.

Imparting Durable Crease-resistance to Cotton. Joseph Bancroft & Sons Co. *BP* 662,683

Crease-resistance without loss in strength is obtained by impregnating the fabric with an aqueous solution of a mixture of a hydroxymethylmelamine (80–20% by wt.) and a methylated hydroxymethylmelamine (20–80%), the strength and pick-up of the solution being such that after drying and baking the fabric contains 3–20% of resin solids. C. O. C.

Modifying Cellulose with Boron Trifluoride. Röhm & Haas Co. *USP* 2,554,439

Aqueous solutions of boron trifluoride dissolve cellulose. Preferably a 58–67% solution is used at − 5 to 20°C., the effect obtained depending on the time of treatment. Thus passing cloth through the solution and immediately washing off results in "chemical singeing", i.e. removal of small fine fibres, loose ends, etc. Slight additional action imparts a linen-like finish. A longer time of treatment renders the fibres more translucent, and organdie effects can be obtained. These border on parchmentising, readily obtained by continuing the action of the aq. BF_3 short of dissolving the fibre. C. O. C.

Rendering Cellulosic Fibres Flame- and Flash-resistant. Lockport Cotton Batting Co. *USP* 2,553,781

The loose fibres are treated in an aqueous solution of borax (1 lb./gal.), boric acid (0·5 lb./gal.), and a surface-active agent (0·1–0·5% on the weight of the soln.) for at least 2 min. at 130–150° F., so that the fibres when dry contain 10–15% of the proofing material. C. O. C.

Catalysts for Accelerating the Cure of Aminoplasts. Monsanto. *BP* 660,366

A hydroxyamine salt formed from an acid and a hydroxyamine of > 3 C in which the NH_2 group is on a C atom adjacent to a C atom to which at least one OH group is attached are latent curing catalysts for non-halogenated aminoplasts, 0·1–5·0 parts per 100 of aminoplast being used. Used in aqueous resin solutions for treating textiles, they remain stable and do not cause premature curing if there is delay in using the solution. The hydrochloride of 2-amino-2-methyl-1-propanol and the phosphate of 2-aminobutanol are suitable for such use. C. O. C.

Rendering Wool Mothproof and Shrink-resistant. American Cyanamid Co. *USP* 2,555,277

The material is treated with a dispersion or solution of a water-dispersible alkylated or "alcohol-reacted" hydroxymethylmelamine containing a fluoride, and is then dried and baked to cure the resin. The mothproof obtained is fast to washing. C. O. C.

Flock Effects on Fabrics. Heberlein & Co. *BP* 662,452

Short-staple fibrous cellulose flock is locally applied to the fabric, and is then fixed by treating the fabric with a solvent for natural or regenerated cellulose so as superficially to dissolve both the flock and the fabric. C. O. C.

Coated Fabrics. DuP. *BP* 662,240

Excellent bonding is secured between cloth and chlorosulphonated polythene by including in the coating composition a polymerised ester whose monomer has the formula $CH_2CX{\cdot}COOY$ (Y = Alk of < 9 C; X = H or CH_3), there being 20–40 parts by weight of the polymerised ester per 100 parts of chlorosulphonated polythene. C. O. C.

Coating Fabrics. Textuff. *BP* 664,157

The fabric is treated with live steam to eliminate absorbed air, uniformly wetted out in an aqueous bath, passed through a bath of a regenerratable cellulose derivative, and finally treated with a coagulant. C. O. C.

Fabrics coated with Polyvinyl Chloride. DuP. *USP* 2,556,885

Better adhesion and flexibility are obtained by interposing between the polyvinyl chloride coating and the base fabric a coating given by an aqueous composition of a butadiene–acrylonitrile copolymer and a water-soluble phenol–formaldehyde thermosetting resin. C. O. C.

Nylon-covered Elastic Yarn. United States Rubber Co. *BP* 661,530

Vulcanised rubber thread is stretched to a selected extent, and coated with nylon from a solution in e.g. formic acid. A strong yarn capable of rapid extension and retraction over at least 50% of its length is produced. J. W. B.

Insect-repellent Fabric. U.S. Secretary of the Army. *USP* 2,555,330

Fabric impregnated with dipyridine—

repels insects, particularly *Aedes aegypti* and *Anopheles quadrimaculatus*. C. O. C.

Treating Carrotted Fur Fibres. American Hatters & Furriers Co., Jonas & Naumburg Corpn., Chapal Donner Corpn., and Pellissier, Jonas & Rivet. *USP* 2,555,883

The susceptibility of carrotted fur fibres to loss by breakage and solubility during the manufacture of felt is reduced by treating them with an aqueous solution containing not < 0·05% of a protein-reactive organic compound, preferably a cationic compound containing > 9 C, e.g. lauryldimethylbenzylammonium chloride. C. O. C.

Rotproofing. N.V. De Bataafsche Petroleum M.S. *BP* 660,434

A solution containing a hydrocarbon solvent, preferably of b.p. < 200°C., a heavy-metal salt of an organic (preferably naphthenic) acid, a small amount of an organic solvent soluble in both oil and water, and an oil-soluble capillary-active substance is used. It rapidly impregnates thick fibrous material even when wet. C. O. C.

Composite Products of Rubber and Textiles resistant to Microorganisms. Dunlop Rubber Co. *BP* 663,273

Composite rubber and textile products which resist attack by microorganisms are obtained by incorporating into the rubber during compounding a hydroxydi(or tri)-arylmethane or a halogen-substituted derivative of such a compound. These compounds resist high temperatures, e.g. during vulcanising, and do not tender the textile. C. O. C.

Non-woven Fabrics. B. M. Glover. *BP* 663,732

Carded web coming from a scribbling machine is laid in superposed layers on the moving platform of a Blamires-type feed. Each layer is sprayed with a mist-like spray of a liquid plastic. The layers are then combined together by means of heat and pressure. C. O. C.

Coating with a Flame Pistol. Schori Metallising Process. *BP* 663,222

When coating with a flame pistol, e.g. of the type described in *BP* 432,831, oxidation of the coating material is avoided by using a non-oxidising gas to convey the powdered coating material. C. O. C.

Apparent Mildew Resistance of Weathered Cotton Duck (VI p. 102).

Coating Regenerated Cellulose Film or other Flexible Non-metallic Surface with Metals (XI p. 105).

XI—PAPER AND OTHER CELLULOSIC PRODUCTS

Isolation and Polymolecularity of Paper Birch Holocellulose. T. E. Timell and E. C. Jahn. *Svensk Papperstidning*, **54**, 831–846 (31 Dec. 1951).

The average degree of polymerisation (D.P.) of the cellulose from paper birch (*Betula papyrifera* Marsh), estimated viscometrically after direct nitration of the wood, was 2975, and the corresponding value for the hemicelluloses (mainly xylan) was 220. The cellulose was found to be very polymolecular, comprising material of D.P. 200–4000 and with a chain length distribution similar to that previously found for cotton linters and cellulose from aspen wood, whereas the xylan exhibited very little polymolecularity. Various methods for isolating holocellulose from wood were compared, and it was found that a modified van Beckum and Ritter method was superior to procedures involving the use of chlorite and permitted the isolation of large amounts of holocellulose within a short space of time. S. V. S.

Oxycellulose. I—Properties of Accelerated Oxidation Oxycellulose. S. H. Mhatre, G. M. Nabar, and G. M. Vyas. *Proc. Indian Acad. Sci.*, **31**, 234–239 (1950): *Chem. Abs.*, **45**, 9856 (10 Nov. 1951).

Investigations of the behaviour of cotton in accelerated oxidations with NaOCl in presence of vat dyes show that—(1) with those vat dyes unaffected by NaOCl, the ratio between reducing and acidic groups in the cellulose was 2; (2) when the dyes were affected by NaOCl, this ratio was 1. These ratios were not affected by conditions of oxidation, e.g. pH. This indicates that the mode of oxidation is not the same in each case. C. O. C.

PATENTS

Treatment of Spent Liquors obtained in the Cooking of Cellulose-containing Materials with Alkali. B. H. E. Gruen. *BP* 662,654

The spent liquor obtained from the digestion of silica-containing straw, grass, bamboo, or other cellulose-containing material with an alkali is treated before evaporation with burnt lime, as precipitating agent, in excess of the quantity equivalent to the silica content of the liquor. The precipitation is carried out at an elevated temperature, e.g. in a closed vessel under pressure, the precipitate separated, and the alkali recovered from the liquor by the usual evaporating, burning, dissolving, and causticising operations. S. V. S.

Paper of High Wet Strength. Monsanto. *BP* 663,717

A product used for imparting high wet strength to paper is obtained by heating in water a mixture of melamine (40–60% by weight) and urea (60–40% by weight) with an aliphatic aldehyde and 0·15–1·5 mol. of an alkali or alkaline-earth metal bisulphite for each mol. of melamine plus urea calculated as melamine. The components may be added to pulp of pH 4–7, and then condensed *in situ* after the paper has been formed by heating it to 210–350°F. C. O. C.

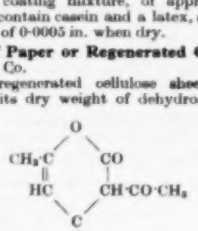
Coating Paper at High Speeds. Time. *USP* 2,555,536

Printing Paper. National Cash Register Co. *BP* 661,523

Printing paper with improved resistance to ink bleed is made by coating with an adsorptive zeolite mineral, e.g. analcite, chabazite, natrolite, heulandite, sodalite, helvite, or noselite, of 10 μ. particle size, mixed with starch as binder. The coating mixture, of approx. 45% solids content, may contain casein and a latex, and is applied to give a coating of 0·0005 in. when dry. S. V. S.

Mildewproof Paper or Regenerated Cellulose. Dow Chemical Co. *BP* 661,473

Paper or regenerated cellulose sheeting containing 0·3–5·0% on its dry weight of dehydroacetic acid—



is mildewproof. C. O. C.

Antiseptic Paper. Interlake Tissue Mills Co. *BP* 663,515

Paper treated with an organic hygroscopic substance, e.g. glycerol, and *o*-hydroxyphenylmercuric chloride or a similar organomercury compound is odourless and remains sterile for a very long time. C. O. C.

Fungicidal Wrapping Paper. Riegel Paper Corpn. *BP* 664,339

Paper is rendered fungicidal by coating it first with a solution of a dialkyl dichlorosuccinate and a filmogen, e.g. ethyl cellulose, in a volatile solvent and then, after drying, with a wax. The succinate vaporises and escapes from the coating only very slowly, and gives protection against mildew to materials wrapped in the coated paper. C. O. C.

Printing and Coating Paper. Champion Paper & Fibre Co. *USP* 2,555,266

A process is described by which in one operation paper is printed, coated, and given a cast surface. C. O. C.

Coating Paper. Time. *USP* 2,556,262

The viscosity or consistency of the coating composition when applied to the web is readily and accurately controlled by supplying the fountain with coating material of very nearly or precisely the same viscosity as that with which it is desired to apply it to the web. The vapour pressure of the atmosphere about the coating material is kept equal to that of the coating material itself, so as to maintain constant viscosity in the coating material as it travels from the fountain to the web. C. O. C.

Imparting Gloss to Paper. United States Rubber Co. *USP* 2,554,662–3

The paper is coated with an aqueous dispersion of an amorphous thermoplastic polymer having a second-order transition temperature between 40° and 125°C. The coated paper is dried at 20–60°C. above this transition temperature, and is then heat-pressed at 40–60°C. above this transition temperature. C. O. C.

Coating Regenerated Cellulose Film or other Flexible Non-metallic Surface with Metals. British American Research. *BP* 663,251

The film is passed through a zone evacuated to a pressure of 50–500 μ. of Hg, where both sides of it are heated so as to drive occluded gases and volatile matter from the film. It then passes into a cooling zone evacuated to a pressure lower than that in the heating zone, there being a vapour barrier between the two zones to prevent the gases evolved from the film from entering the cooling zone and condensing on the substrate. The film is cooled at least 20°C. by passing it against at least one cooling surface in the cooling zone, so as to prevent further evolution of gases from the film. The time the film is in these heating and cooling zones is such that it does not become embrittled. From the cooling zone the film passes into a coating zone evacuated to below 0·5 μ. of Hg, where the metal is vaporised and condensed on the film. C. O. C.

Vinylidene Chloride–Acrylonitrile Copolymers as Moistureproof Coating for Sheet Materials. British Cellophane. *BP* 663,645

Non-fibrous organic sheets can be satisfactorily given a moistureproof coating with a vinylidene chloride–acrylonitrile copolymer by offering an aqueous dispersion of the copolymer simultaneously to two surfaces of the organic sheet in the form of a nip of controlled width and then separating the two sheets before they have had time to absorb water from the dispersion. Each of the sheets then bears a liquid coating of predetermined thickness and of sufficiently low viscosity to level itself to a smooth coating. C. O. C.

Smudge-resistant Transfer Sheets, Typewriter Ribbons, etc. Ditto. *USP* 2,554,909

Typewriter ribbons, carbon papers, etc. coated with a water- and alcohol-soluble basic-dye-containing composition are surface-treated with a substance, e.g. ammonia or a polyamine containing > 1 N each having at least one H atom attached to N, which converts the dye to its water-insoluble dye base and which also, by itself or in conjunction with a reducing agent, reduces the dye base to the even more insoluble and colourless leuco base. This gives effective protection against smudging. C. O. C.

Pressure-sensitive Adhesive Tape. Minnesota Mining & Manufacturing Co. *BP* 663,036

Pressure-sensitive adhesive tape is manufactured by forming a backing by impregnating and unifying a soft,

flexible porous paper with a rubberlike butadiene–acrylonitrile (20–50 : 80–50 parts) copolymer, and then coating the backing with a layer of a normally tacky pressure-sensitive adhesive. The first step is to apply an aqueous dispersion of the copolymer, containing 30% total solids, to give 3 grains of copolymer per 24 sq. in. after drying, the second step being to apply the equivalent of 7–9 grains per 24 sq. in. Drying follows both steps. S. V. S.

XII—LEATHER; FURS; OTHER PROTEIN MATERIALS

Lime Sensitivity of Anionic Leather Dyes. G. Otto. *Leder*, **2**, 210 (1951); *J. Soc. Leather Trades Chem.*, **35**, 388 (Nov. 1951).

Tanneries mostly use mains water in making up the float for dyeing; as water in Germany is usually hard, there is interaction between the lime and the dye. With basic dyes, it is merely the alkalinity that is objectionable; this can be overcome by the addition of acetic acid. With anionic (acid and substantive) dyes, the anions often react with the calcium cation to form compounds of low solubility. The reaction may be so strong that cloudy precipitates are formed which do not dye. Also, when these precipitates form only a small part of the total dye, they may accumulate in those parts of the leather which have a looser structure, i.e. the belly, causing them to be darker and sadder; the flesh side tends also to absorb the precipitated dye. In certain cases the interaction between dye and lime is of advantage, giving uniform and bodied colour. Tests have been carried out on the interaction between anionic dyes and calcium salts. To do this, it is necessary to standardise conditions; the use of $CaCO_3$ was adopted, prepared by passing CO_2 through a 5% lime suspension until the pH reached 7·6. The coarse precipitate was removed by decantation, and the fine particles were allowed to settle. A dispersion of these was homogenised by supersonic radiation; it was used in two concentrations—74 and 10 g. CaO per litre.

Solutions of 8 acid and 14 substantive dyes were dissolved in water to give a concentration of 1/15,000 M.; to 10 ml. of solution, 1 ml. of $CaCO_3$ dispersion was added, shaken, and allowed to stand. The colour of the clear solution was determined; the results are given in four tables, showing dyes that do not react with $CaCO_3$ and those that react slightly, more strongly, and very strongly. It is observed that in those dyes which do not react the aromatic nucleus is strongly negative owing to the presence of sulpho or nitro groups. Reaction occurs when—(*a*) A positive nucleus carries two amino groups not balanced by a sulpho group. (*b*) An aromatic nucleus has two OH groups. (*c*) A naphthalene nucleus has an amino group. The reaction is especially strong in the presence of COOH when it is (*d*) in the form of a salicylic acid residue, i.e. in the *ortho* position to a phenolic OH; (*e*) as a phenylglycine residue. In practice, only those dyes that react strongly with lime would be avoided; those of medium reaction often dye well in a water of medium hardness, as they give uniform coloration and good cover. C. J. W. H.

PATENTS

Leather Substitute. S. S. Royko. *BP* 663,758

Waste chrome leather (except pig leather) cuttings are beaten into a smooth pulp containing 45 g. dry fibre per litre. After addition of grease or oil to the pulp it is made alkaline, and then blood, natural or synthetic rubber latex, and a vulcanising accelerator are added followed by Na_2S, S, colophony, a dye, $(NH_4)_2SO_4$, H·CHO, H_2SO_4, and NH_4OH. Finally the material is pressed into sheets, dried, vulcanised, and coated with a casein or cellulose nitrate dressing. C. O. C.

XIII—RUBBER; RESINS; PLASTICS

PATENTS

Methylal as Solvent or Softener for Polyvinyl Compounds. Celanese Corpn. of America. *BP* 663,148

Methylal is an excellent solvent for polystyrene, polyvinyl acetate, polymethyl acrylate, polyvinyl formals, acetals and butyrals, and butadiene–styrene copolymers. It is not a solvent for homopolymers of vinyl compounds containing halogen, e.g. polyvinyl chloride. C. O. C.

Stabilising Synthetic Resins. Societe Anonyme des Manufactures des Glaces et Produits Chimiques de Saint-Gobain, Chauny et Cirey. *BP* 664,142

Polymers and copolymers of halogenated derivatives of ethylene are stabilised and given improved mechanical properties by adding to them 0·5–2·0% by weight of an alkali-metal or magnesium thiophosphate. C. O. C.

Ornamented Embossed Articles. G. W. Borkland. *BP* 664,167

A sheet of thermoplastic material is heat-embossed and cold-set. A decorative coating is then applied to selected portions of the embossed areas, after which the material is heated and reformed into a flat sheet, when the applied coating becomes distorted. This distorted pattern is then reproduced on a second sheet of the thermoplastic material, e.g. by photographing it on to a printing plate, which is used to print the design on the second sheet, after which the sheet is embossed to produce the original embossed pattern on it. C. O. C.

Catalysts for Accelerating the Cure of Aminoplasts (X p. 104).

Vinylidene Chloride–Acrylonitrile Copolymers as Moisture-proof Coating for Sheet Materials (XI p. 105).

XIV—ANALYSIS; TESTING; APPARATUS

Fourth Annual Review of Analytical Chemistry. *Anal. Chem.*, **24**, 2–131 (Jan. 1952).

Twenty-four individual headings include—amperometric titrations, chromatography and analogous differential migration methods, distillation, ion exchange, extraction, organic microchemistry, inorganic microchemistry, inorganic gravimetric analysis, inorganic volumetric analysis, volumetric analytical methods for organic compounds, characterisation of organic compounds, and experimental statistics. J. W. D.

Automatic Operations in Analytical Chemistry. G. D. Patterson and M. G. Mellon. *Anal. Chem.*, **24**, 131–143 (Jan. 1952).

A review of 12 months' progress in the automatisation of chemical analysis, with 287 references to the literature. J. W. D.

Standard Tests for Wetting Agents—Estimation of Wetting-out Properties. German Standard *DIN* 53901. *Textil-Praxis*, **6**, 863 (Dec. 1951).

An apparatus is described for assessing the wetting-out power of wetting agents. A piece of cotton material is fixed in a circular metallic frame attached to a thread of Perlon and placed in a cylindrical vessel containing water. The time taken from entering the material till it begins to sink is taken as a measure of the wetting-out power. Tests are carried out in distilled water and in water of standard hardness. B. K.

Mechanism of the Wetting of Textiles. I. J. Gruntfest. *Text. Research J.*, **21**, 861–866 (Dec. 1951).

The use of a hydrometer method for determining data on the wetting of textiles is discussed. The effects of rate of immersion, skin density, solution temperature, and mechanical action are described. The time dependence of the surface tension of solutions of surface-active agents is discussed, and it is shown that the rates of surface-tension lowering correlate quite well with wetting agent performance. A. B.

Improvement in Anthrone Method for Determination of Carbohydrates. F. A. Loewus. *Anal. Chem.*, **24**, 219 (Jan. 1952).

Difficulty caused by discoloration of the reagent (usually 0·1–0·2% anthrone in 95% H_2SO_4) is overcome by using the anthrone in ethyl acetate soln., in which it is stable for some weeks. J. W. D.

Perchloric Acid Method for Determination of Starch and some Practical Applications. W. G. Sink and D. L. Worth. *Amer. Dyestuff Rep.*, **40**, P 848–P 850, P 866 (24 Dec. 1951).

The following modification of McEwen's colorimetric method for starch determination is presented—Fabric samples (5–10 g.) containing residual size and of known dry weight are cut into small squares, covered with approx. 30 ml. of 42% perchloric acid in a 100-ml. beaker, and allowed to stand at room temperature for 30 min. with

frequent stirring. 100 ml. of distilled water and a few drops of phenolphthalein are added, followed by 6 N-NaOH until the solution turns pink, which is then discharged by 2 N. acetic acid. The solution is filtered and made up to 250 ml. with distilled water. An aliquot containing 1·5–3·5 mg. starch is made up to about 50 ml., and 2·5 ml. of 2 N. acetic acid, 0·5 ml. of 10% KI soln., and 2 ml. of 0·05 N-KIO_3 are accurately added, and the solution is diluted to 100 ml., shaken, and allowed to stand for 10 min. It is compared colorimetrically with a standard solution of similar starch. Urea–formaldehyde resins and fats do not interfere. J. W. B.

Coagulation of Soluble Polysaccharides and other High Polymers by Electrolytes. H. Deuel and J. Solms. *Kolloid-Z.*, **124**, 65–69 (Nov. 1951).

Tables are given of the precipitation values of 36 water-soluble polysaccharides and other high polymers by 25 different electrolytes. It is suggested that these precipitation reactions can be adapted as qualitative tests for these compounds. L. P.

Chemical Analytical Study of Salicylanilide. I—Differential Reactions of Salicylanilide, Salicylamide, and *N*-Acetylsalicylanilide. G. Matta and M. L. Nunes. *Anais Azevedos* (Lisbon), **3**, 21–36 (1951): *Chem. Abs.*, **45**, 10,143 (25 Nov. 1951).

Commercial salicylanilide (I) can be prepared by dissolving it in cold 10% NaOH and precipitating with 10% HCl; it can be determined volumetrically by titrating with 0·1 N-NaOH in alcohol using a phenolphthalein indicator. The refractive index n_D at m.p. is 1·59 for I, 1·55 for salicylamide (II), and 1·535 for *N*-acetylsalicylanilide (III). The following reactions distinguish between the three compounds—Nitration: I purple, II yellow, III yellow ppt.; aq. $FeCl_3$: I salmon pink ppt., II yellow, III red; aq. $AgNO_3$: I white ppt., II yellow ppt., III no reaction; aq. Co acetate: I light orchid ppt., II red–violet, III orange–yellow ppt. C. O. C.

Determination of Anthracene and Cadmium as Cadmium Anthracenesuccinate. I. Ubaldini and F. Guerrieri. *Ann. Chim.* (Rome), **41**, 247–254 (1951): *Chem. Abs.*, **45**, 10,143 (25 Nov. 1951).

An aq. crystalline salt insoluble in water forms rapidly $(C_{14}H_{12}O_4Cd,2H_2O)$ when Cd^{++} is added to a soln. containing the anthracenesuccinate ion at about pH 7. This serves for determining either anthracene or Cd as well as for separating Cd from Cu. C. O. C.

Effect of Gelatin on the Polarographic Waves of *p*- and *o*-Nitroanilines. N. Tanaka and M. Kobayashi. *Bull. Chem. Soc. Japan*, **24**, 132–135 (July 1951).

p- and *o*-Nitroanilines give polarographic waves with distinct maxima. These maxima are suppressed by the addition of 0·005–0·01% gelatin to the supporting electrolyte, but above this concentration of gelatin the waves are split into two distinct waves, whose relative magnitude depends on the concentration of gelatin. The magnitude of the effect is greater for *p*-nitroaniline than for *o*-nitroaniline, and this difference has been used for the quantitative estimation of mixtures of the two nitroanilines by the solution of two simultaneous equations. The results obtained by varying known mixtures in ratios from 1 : 5 to 5 : 1 are satisfactory. W. J. M.

Substituted Benzidines and Related Compounds as Reagents in Analytical Chemistry. VIII—3 : 3′-Dimethylnaphthidine as Indicator in the Titration of Cadmium, Calcium, and Indium with Ferrocyanide. R. Belcher, A. J. Nutten, and W. I. Stephen. *J.C.S.*, 3444–3447 (Dec. 1951).

Chromatographic Properties of Oil-soluble Coal-tar Colours. L. C. Weiss. *J. Assocn. Off. Agric. Chem.*, **34**, 453–459 (1951): *Chem. Abs.*, **45**, 9865 (10 Nov. 1951).

The behaviour is described of 22 oil-soluble dyes, 17 certifiable and 5 non-permitted (in U.S.A.), on various chromatographic adsorbents with various solvent systems. The data indicate that with appropriate adsorbents and solvents many of these dyes can be separated from each other chromatographically. C. O. C.

Determination of Unsulphonated Phenolic Intermediates in Dyes. H. Holtzman and H. Graham. *J. Assocn. Off. Agric. Chem.*, **34**, 393–395 (1951): *Chem. Abs.*, **45**, 9865 (10 Nov. 1951).

Collaborative results are given for a method of determining β-naphthol in D&C Red No. 9 and D&C Red No. 11. This method, previously found satisfactory for β-naphthol in D&C Red No. 35 and D&C Orange No. 17, involves extraction with petroleum ether, coupling with diazotised sulphanilic acid, and reduction of the resulting dye with $TiCl_3$. C. O. C.

Subsidiary Dyes in FD&C Colours—Determination of Lower Sulphonated Dyes in FD&C Yellow No. 5. M. Dolinsky. *J. Assocn. Off. Agric. Chem.*, **34**, 411–416 (1951): *Chem. Abs.*, **45**, 9865 (10 Nov. 1951).

The method given in *A.O.A.C. Methods of Analysis* (6th edition 1945) has been modified to allow spectrophotometric determination of the subsidiary dye. This modification reduces the amount of sample required and increases the accuracy of the determination. Two monosulphonated subsidiary dyes of FD&C Yellow No. 5 were prepared, and their spectrophotometric characteristics determined. C. O. C.

Determination of Arsenic and Antimony in Dyes. L. S. Harrow. *J. Assocn. Off. Agric. Chem.*, **34**, 396–404 (1951): *Chem. Abs.*, **45**, 9865 (10 Nov. 1951).

The Cassil–Wichmann method (*Chem. Abs.*, **33**, 6068 (1939)) is satisfactory for determining As in dyes if the apparatus is modified to include two absorbers in series. Sb can be determined by a modified Cassil–Wichmann procedure in which Zn and HCl are substituted for $SnCl_2$ and KI. A preliminary separation of the two metals is necessary for determining As in presence of Sb. C. O. C.

Determination of Cadmium in Dyes. C. Stein. *J. Assocn. Off. Agric. Chem.*, **34**, 417–421 (1951): *Chem. Abs.*, **45**, 9865 (10 Nov. 1951).

A dithizone method for determining Cd in dyes is described. C. O. C.

Determination of Copper in Dyes. C. Stein. *J. Assocn. Off. Agric. Chem.*, **34**, 427–432 (1951): *Chem. Abs.*, **45**, 9865 (10 Nov. 1951).

Greenleaf's method (*Chem. Abs.*, **36**, 4608 (1942)) has been modified to simplify the procedure and reduce the blank. Cu is isolated by dithizone and determined with Na diethyldithiocarbamate. C. O. C.

Determination of Lead and Bismuth in Dyes. C. Stein. *J. Assocn. Off. Agric. Chem.*, **34**, 422–427 (1951): *Chem. Abs.*, **45**, 9865 (10 Nov. 1951).

A dithizone method for separating and determining microquantities of Pb and Bi in dyes is described. Pb is determined colorimetrically or by electrolysis and titration; Bi is determined colorimetrically. C. O. C.

Determination of Intermediates derived from Phthalic Acid. C. Graichen. *J. Assocn. Off. Agric. Chem.*, **34**, 407–411 (1951): *Chem. Abs.*, **45**, 9866 (10 Nov. 1951).

D&C Yellow No. 10 is dissolved in aqueous acid and extracted with ether. The ether extract is in turn extracted with dil. alkali, and the phthalic acid extracted by the alkali determined spectrophotometrically. Phthalic acid in D&C Red No. 19 and D&C Yellow No. 11 is determined similarly, except that the dye is dissolved in chloroform for extraction with dil. alkali. Collaborative study has shown that these methods, although not of great precision or accuracy, are satisfactory for determining phthalic acid in the amounts ordinarily found in the above three dyes. C. O. C.

Micro Methods in the Paint and Lacquer Laboratory. III—Determination of Lead in Dry Materials. H. Flaschka and H. Lockner. *Fette und Seifen*, **53**, 80–81 (1951): *Chem. Abs.*, **45**, 9888 (10 Nov. 1951).

Precipitation of Pb (II) with a benzene solution of thioacetamide is followed by centrifuging, dissolving the PbS in HNO_3, neutralising with NH_4OH, acidifying with acetic acid, precipitating with $K_2Cr_2O_7$, filtering, and finally determining the CrO_4^{--} volumetrically. C. O. C.

Cobalt Analysis in Inks and Driers. A. C. Zettlemoyer and W. C. Walker. *Amer. Ink Maker*, **28**, (10), 69, 71, 101 (1950): *Chem. Abs.*, **45**, 10,609 (25 Nov. 1951).

A rapid, very accurate method for the colorimetric determination of Co in inks and driers is—(1) weigh the

ink sample in a centrifuge tube; (2) dissolve the ink in petroleum ether, and separate the pigment by centrifuging; (3) determine the colorimeter calibration curve with a series of solutions of known Co concentration; (4) extend the solution with conc. HCl; and (5) examine the acid solution in the colorimeter. C. O. C.

Cotton Fineness and Immaturity as measured by the Arealometer. K. L. Hertel and C. J. Craven. *Text. Research J.*, **21**, 765–774 (Nov. 1951).

An air-flow instrument, the arealometer, is described, which is dependent upon the surface area and the immaturity of cotton. The calibration of the instrument is described, and it is concluded that the method provides a quick measure of both fineness and immaturity from two readings on the arealometer. A. B.

Centrifugal Method of Measuring Fibre Swelling. J. M. Preston and M. V. Nimkar. *Bull. Inst. textile France*, (18), 53–58 (1950): *Chem. Abs.*, **45**, 9867 (10 Nov. 1951).

Centrifugal techniques for measuring fibre swelling measure both the water of swelling (in the fibres) and capillary water (between the fibres). Capillary water can be reduced, and more consistent results obtained, by adding a surface-active agent to the water used. Total water retained is 1–10% lower at surface tension 36 dynes/cm. than at 73 dynes/cm. except for calcium alginate fibres, where it is higher. C. O. C.

Colour Measurement and its Application in the Textile Industry. H. J. Selling. *Textil-Rund.*, **6**, 498–508 (Nov. 1951).

The relation between the X, Y, and Z values of dyeings produced from a red, a yellow, and a blue dye and the concentrations of the dye components is given by equations due to Kubelka and Munk. Solution of the equations algebraically is tedious. A simpler graphical method, which is suggested for the prediction of dyeing recipes, consists in the superimposition of three diagrams on which are plotted the amounts of the dye components required to give the X, Y, and Z values of the pattern to be matched. Various formulæ which have been proposed for expressing degree of whiteness in a single figure are reviewed, and for a series of paper samples the figures given by the various formulæ are compared with visual estimates of whiteness. F. A.

Determination of the "Whiteness" of Textile Materials. L. I. Belenky and M. E. Kazanskaya. *Tekstil. Prom.*, **11**, 23–25 (Oct. 1951).

The average observer can distinguish a difference in degree of whiteness corresponding to a difference of about 0·5% in the reflectance coeff. The existing visual photometers enable this coeff. to be measured with an accuracy of ± 1–4%, depending on the observer and on the variation in the spectral characteristics of the white. A description is given of a simple differential photoelectric colorimeter that has been found highly satisfactory for whiteness measurements. Light from a 100-watt filament lamp on a const. voltage supply passes through a narrow-waveband filter, through a central hole in a selenium photocell, and on to the specimen or standard white surface. Reflected light is received on the annular surface of the photocell. The lamp illuminates also a second photocell, which can be rotated about a diameter, thus enabling the total incident radiation to be varied. The responses of the cells are opposed through a potentiometer system incorporating a sensitive galvanometer as a null instrument. The arrangement gives results that are identical with those obtained with two types of photospectrometer, the accuracy in each case being about ± 0·2%. The results of determinations on a number of fabrics are given, and the effects of fabric construction and lustre are discussed. A. E. S.

Comparison of Direct Colorimetry of Titanium Pigments with their Indirect Colorimetry based on Spectrophotometry and a Standard Observer. D. B. Judd. *J. Optical Soc. Amer.*, **39**, 945–950 (1949).

A "Colour Index" for Floor Tile Resins. B. A. Reeves. *A.S.T.M. Bull.*, (178), 62–64 (Dec. 1951).

The visual measurement of the colour of resins and other components of floor tiles by the coal-tar colour number method is difficult and unreliable. The use of a photoelectric photometer is suggested, and the transmittance of a benzene soln. of the resin is compared with that of water, the instrument being first calibrated against a freshly prepared set of coal-tar resin colour standards. Colour ratings obtained by using the instrument are frequently darker than by visual inspection, but are considered to be more reliable. A. J.

New Photoelectric Prismatic Colorimeter. F. Frauzon and H. Goden. *Z. ges. exptl. Med.*, **117**, 430–438 (1951): *Chem. Abs.*, **46**, 4 (10 Jan. 1952).

Light from a uniform source is passed through a cell containing the unknown to an Se photoelectric cell and through a prism to another Se cell. The opposite poles of the Se cells are balanced through a null galvanometer by moving the prism. The calibration curve of the instrument is a straight line. C. O. C.

Experimental Colour Comparator. E. J. King and D. S. Rodbell. *J. Optical Soc. Amer.*, **41**, 830–835 (1951): *Chem. Abs.*, **46**, 4 (10 Jan. 1952).

The design and construction of an experimental colour comparator, which permits binocular viewing of a constant two-degree split matching field with a dark surround, are discussed. During observation of the field, continuous adjustments of the colour of the "standard" side are possible with three controls, each associated with one of the subjective attributes of colour. Data useful in calculating the C.I.E. tristimulus values for the matched sample appear on calibrated dials. Colour samples to be matched by Illuminant "C" are inserted in the instrument on small cards. Larger samples, external to the comparator, e.g. textiles, can be evaluated for all types of illumination. In every case texture in the sample is observed by the comparator mechanism. Date are given to indicate the matching tolerances that can be expected. C. O. C.

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Microscopical Dyeing Phenomena; Studies with the Microdyeoscope (VIII p. 102).

XV—MISCELLANEOUS

Coloured Gold Plating. E. R. Thews. *Metal Finishing*, **49**, (9), 80–85 (1951): *Chem. Abs.*, **45**, 9396 (10 Nov. 1951).

Coloured gold plating can be produced by—(1) deposition of coloured alloys; (2) deposition of very light, translucent gold films on a coloured undercoat; (3) alloying of deposited gold films with the undercoat or with previously or subsequently deposited films of other metals by diffusion; (4) etching coloured gold alloy deposits to remove one or the other base metal. A green deposit is obtained with a bath containing gold chloride (22 g./gal.) and silver chloride (7·5) or from a double cyanide bath containing also NaCN, Na_2CO_3, Na_3PO_4, and Na_2SO_4. An antique green is given by a bath containing gold chloride (7·5), AgCl (0·5), As (0·2), and KCN (85·9), or from a bath containing NaAu cyanide (15), CaO (2·5), Pb acetate (1), and NaCN (30). A pink plating is given by NiAu cyanide (25), NaCN (12–15), and NaOH (110–125). Other methods of producing coloured deposits are also described. C. O. C.

Evolution of a Patent—A Symposium. *Ind. Eng. Chem.*, **43**, 2487–2496 (Nov. 1951).

The papers deal with the following aspects of patent procedure—transforming ideas into patent property, a technical-patent liaison group, selection of patent counsel, invention records, reporting invention data, and chemical patent searches. The introductory paper stresses the importance not only of research and development work but of the patents which protect this work. A. H.

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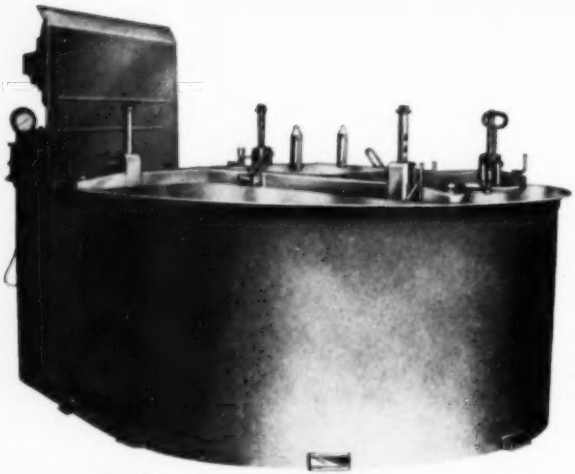

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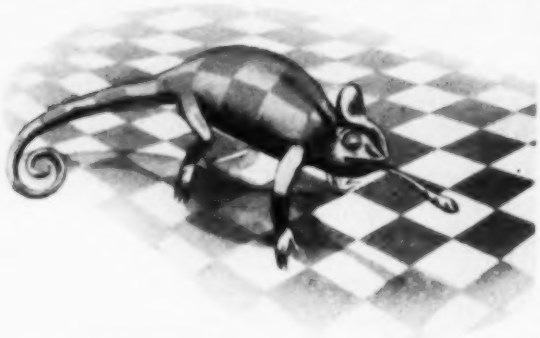

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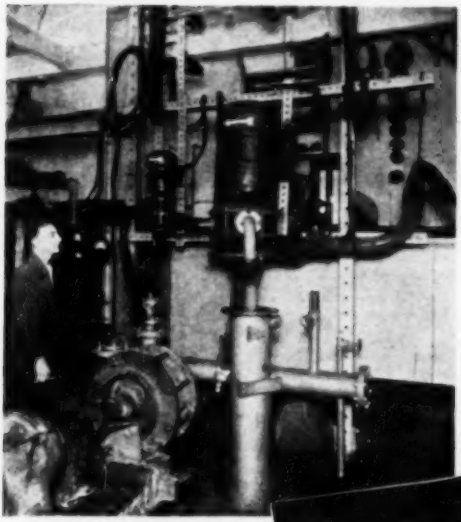

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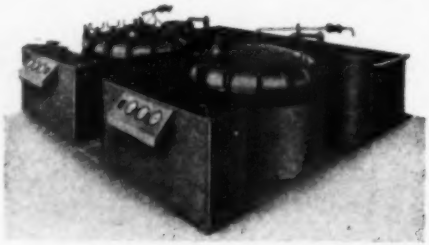

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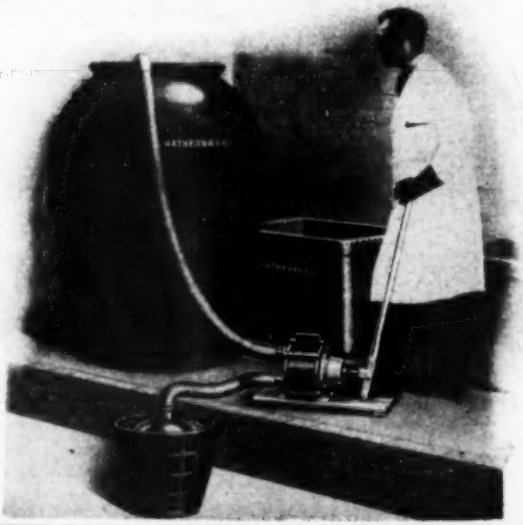

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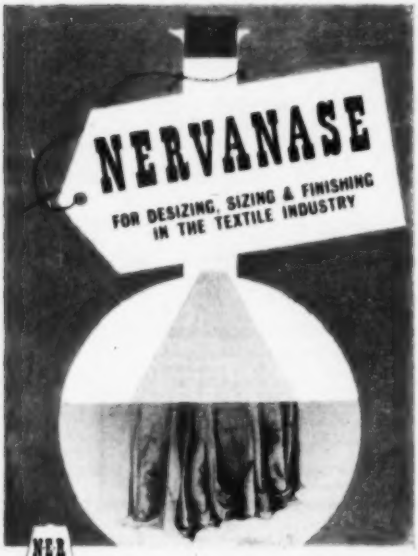

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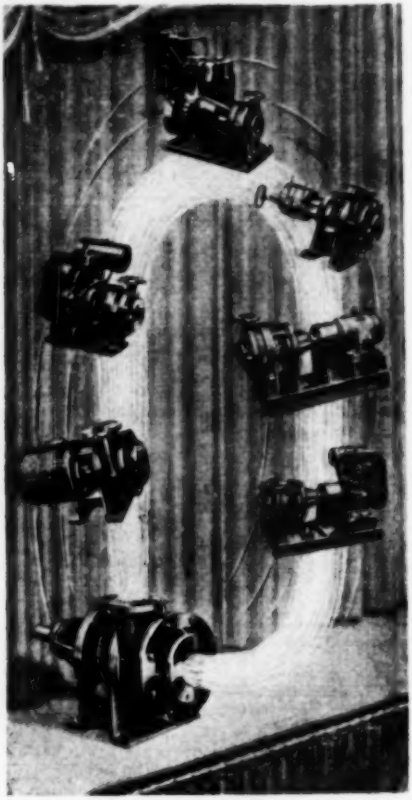

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